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AERONAUTICAL STRUCTURES LABORATORY

REPORT NO. NAVAIRMATCEN-ASL-1050, PART V
DATE: 10 APRIL 1962

DEVELOPMENT OF HIGH-TEMPERATURE STRAIN-GAGE
BONDING MATERIALS CURABLE AT LOW TEMPERATURES
JULY 1961

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CHICAGO, ILLINOIS
CONTRACT NO. N156-39304

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FOREWORD

This report was prepared by the Ceramics Research Division of the Armour Research Foundation of Illinois Institute of Technology under Navy contract No. N156-39304 and is a continuation of the work performed under contracts Nos. N156-37982 and N156S-32317. Contract No. N156-39304 was initiated by Richard Friedman under Problem Assignment 1-23-8 (formerly project RAAD-298.1), "Development of High Temperature Strain Gages." The work was administered under the direction of the Aeronautical Structures Laboratory, Naval Air Material Center, with Charles R. Nelson acting as project engineer.

This is the final report in this series and covers work performed from February 1960 to July 1961. Work performed under contract No. N156-37982 (November 1958 to February 1960) is covered in report No. NAVAIRMATCEN-ASL-1050, Part IV, and work performed under contract No. N156S-32317 (June 1955 to November 1958) is covered in reports Nos. NAVAIRMATCEN-ASL-1050, Parts I, II, and III.

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PUBLICATION REVIEW

This report has been reviewed and is approved.

R. W. SHACKFORD, Commander, USN
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ABSTRACT

Investigations have been conducted to develop a high-temperature
strain-gage cement which can be cured at low temperatures. Various com-
pounds were incorporated into a basic formulation consisting of silica,
alumina, chromic anhydride, monoaluminum dihydrogen phosphate, and Goulac
suspending agent. The most satisfactory additive was yttrium nitrate
hexahydrate which yields a composition, B144, with good curing at 350°F,
excellent electrical behavior to 1600°F, and moderate strengths. Data
on properties of cements containing other yttrium compounds, as well as
zinc and magnesium compounds, are included in this report.

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FOR ELECTRICAL STRAIN GAGES

I. INTRODUCTION

As temperature requirements increase for missile air-frames, difficulties arise not only in the selection of structural materials but also in the testing of these materials under environmental conditions. This is especially true in the case of utilization of strain gages at elevated temperatures; increased conduction in the cement and thermally induced electric currents result in uncertainty in strain measurements, and therefore in structural performance.

The past year represents the third on this program and also the third change in objective. Previously, efforts were directed to the development of two distinct strain gage cements. One was to have adequate curing properties at below 220°F and to be useful to 800°F, and the other was to be cured below 450°F and suitable for use to at least 1600°F. These objectives were substantially met although presently available strain gages are not reliable at the higher temperatures and do not permit actual application testing of the experimental cements. In the phase just completed, the two separate objectives were combined into one to produce a cement that could be cured below 220°F (preferably room temperature) and be useful to 1600°F.

II. EXPERIMENTAL PROCEDURE

A. Introduction

Throughout this program, tests were designed primarily for speed and simplicity while retaining effective evaluation of the pertinent properties of experimental cements to determine their suitability for strain gage applications. The enormous number of formulations and heat treatments required for a comprehensive survey of inorganic binder systems necessitated a fairly rapid screening program.

The actual mounting of strain gages was not seriously considered for screening tests. Their cost, difficulty in mounting, variability in properties, and problems in measurement would impose severe limitations on the program. In the previous phases, some strain gages were mounted and tested with experimental cements, as well as commercial cements, to evaluate their comparative ability to transmit strain. Results were generally inconclusive and this type of testing was not considered in this phase.

The desirable properties in a strain gage cement are:

1. Adequate electrical resistivity over the desired temperature range to prevent shunting between the strain gage legs and the basis metal,

2. Sufficient strength and bonding at all temperatures to transmit strains,

3. Ability to thermal cycle without loss of bond,

4. Ability to withstand very rapid heating without loss of electrical or bonding properties,

5. Moisture resistance at room temperature,

6. Inertness to gage and substrate alloys at all temperatures.

B. General Considerations

It has been observed that simple electrical resistance measurements across two bonded metal members correlates well with strain gage-to-ground resistances measured by the Aeronautical Structures Laboratory. The observed resistance in megohms is then more meaningful than conversions to volume resistivity based on geometric considerations. The observed resistances are primarily interface effects due to polarization of mobile ions in the cement and are influenced by the degree of contact between cement and metal. Generally, weakly bonded cements offer more contact resistance and therefore higher resistivity, but at an anticipated sacrifice of strain transmission. The most useful strain gage cements may require a compromise between electrical and strength properties.

When resistances through the cement film are relatively low or when moisture is still present, an upward drift in the ohmmeter reading is observed. This is probably due to a barrier layer of ions which migrate more rapidly than can be discharged at the interface, thus repelling further

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ion migration and consequently causing an increase in measured resistance. Migrating ionic species are probably hydrogen or hydronium and hydroxyl which cannot be removed completely from inorganic cement systems except at extremely high temperatures. After an effective cure, enough of the ions are removed through volatilization to eliminate the ohmic drift, but the unremovable portion increases in mobility at higher temperatures so that there is a marked resistance decrease with increased temperature. The presence of the polarizable ions poses an instrumentation problem in that resistance readings become a function of temperature, heating rate, and the voltage applied.

The purpose of the program could then be stated as the selection of the inorganic binder system with best electrical properties at 1600°F and the development of compositions which will permit minimization of ionizable materials at the lowest attainable temperature. Upon attaining minimum hydrate content, the electrical properties for a particular binder system probably cannot be significantly improved if all materials employed are initially of highest purity. Therefore, the approach to this phase of the program was mainly a continuation of the previous phase, i.e., the investigation of the effects of additives and special treatments on curing temperature.

The testing program was designed to determine the curing temperature and time, tensile strength, and thermal cycling properties of experimental compositions. Relatively simple tests were devised which quite comprehensively evaluated the compositions.

C. Specimen Preparation

In the previous phase, bonded overlapping inconel strips were used as specimens. These were adequate for evaluation of the cements, but a simpler configuration was desired. For this purpose, one-half inch diameter inconel cylinders, one-half inch thick were prepared. A three-quarter inch hole tapped with a 1/4 x 28 thread in the center served to permit a central exit for volatiles on baking and also insertion of a one-quarter inch inconel rod for electrical and strength measurements. On one face, two eighth-inch holes were drilled to within one-eighth inch of the opposite face for thermocouple insertion. The cement was applied to the face without

the thermocouple wells.

The experimental cement was applied in a thin film to one or both sandblasted faces of the specimen and allowed to dry. This base coat prevented metal-to-metal contact. The bonding coat was then applied to one face and the two cylinders comprising a specimen were squeezed together (see Figure 1). Excess cement was wiped off and the specimen submitted to a curing cycle, usually consisting of an air set, an oven bake, and a final cure at required temperature.

The height of the cylinders and cement thickness were accurately determined utilizing a micrometer. As electrical properties were not dependent upon thickness (because the matrix of the cement is far more conductive than at the interfaces) this value is not reported in the tables, but is recorded for every specimen in the laboratory logbooks. Generally, thin cement films displayed somewhat higher tensile strengths than thicker films, but other variables were more important.

D. Electrical Testing

For resistance measurements during heating or while curing, two lengths of threaded inconel rod were screwed into the ends of the specimen. The assembly was placed in a nichrome wound split tube furnace operated at 110 volts. A chromel-alumel thermocouple was inserted into one end of the specimen and was connected to a recorder-controller through which the furnace was operated. A voltage controller was used for maintaining curing temperature with full voltage employed for actual testing. Some testing was conducted to 900°F which was attained in eight to nine minutes and others to 1600°F, attained in 23 to 30 minutes. The rods extending outside the furnace were connected to a vacuum tube voltmeter (VTVM) (see Figure 2).

Resistance was observed at room temperature and at 50 or 100°F increments by switching the meter from a voltage to the ohms scale. In this way the initial resistance could be determined, as well as the drift due to ion migration and concentration. If the meter were left on the resistance scale, only the high equilibrium readings could be obtained. Upon reaching test temperature, some resistances were obtained on cooling. The cooling cycle should exhibit higher resistivity because of the heat treatment;

however high temperature reactions, such as corrosion, can introduce conductive products.

The electrical data is plotted on semi-log paper as the logarithm of the resistance versus temperature. In ideal systems, the plot against reciprocal absolute temperature would yield a straight line, but as this does not occur with these compositions, straight temperature is more convenient. Improper curing is displayed as a dip in the curve. The temperature at the minimum of the dip is the approximate minimum curing temperature required to eliminate resistance drops and recoveries on heating.

A quartz tube furnace was constructed for more rapid heating and is shown in Figure 3. The 110 volt, 500 watt G. E. quartz lamps were inserted into transite walls. The other walls were of aluminum to reflect the radiation. Here the specimen is mounted vertically and measured in the same manner as with the split tube furnace, except that the VTVM must be left on the ohms scale as the rapidity of testing does not permit the removal of the charge distribution between readings. The use of four lamps at 110 volts permitted a temperature rise to 900°F in about four minutes.

As the resistance decreased with increasing temperature, the question arises as to what is the limiting minimum permissible resistance. It is assumed that a resistance of less than 10,000 ohms may influence readings on a 120 ohm gage. If one-hundred gages are mounted on a metal component, they can be considered in parallel and the required cement resistance would be 100 x 10,000 ohms or one megohm. As 100 gages is a reasonable number for a strain gage installation, an objective of this program became the maintenance of at least one megohm resistance to as high a temperature as possible. Furthermore, it was considered that temperatures at which the resistance dropped below 0.3 to 0.6 megohm was the absolute limit of usefulness for normal applications. Of course, if fewer gages are used, even higher temperatures might be suitable.

E. Strength Tests

After curing or testing, the specimen with rods attached was placed in a Dillon Tensile Tester and broken in tension (see Figure 4). The cemented area was approximately 0.39-inches² and tensile strength in psi was calculated by multiplying the indicated load by two and one-half. Also

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recorded was whether breakage occurred in adhesion (between metal and cement) or in cohesion (within the cement). Typical breaks are shown in Figure 5.

In the previous phase, strengths were determined in tensile shear. It was found that straight tensile strengths were higher.

The major purposes in measuring strength is to gain insight into the effects of additives and to evaluate the thermal cycling properties of the cement. The actual strength requirement of a strain gage cement has not been determined. A relatively weak commercial cement, Allen P1, appears to adequately transmit strains wherever used, so the strength criterion in this phase was that of Allen P1. If the best electrical cements prove too weak structurally, alternative compositions are presented in this report for high strength with quite good electrical properties.

F. Vacuum Curing

Attempts were made to reduce curing temperatures by use of vacuum. Vacuum curing was performed by inserting the specimen in a Pyrex tube, one end of which was drawn out to a connector tube for attachment to a vacuum pump. The other end, large enough to accept samples, was connected by a ground glass joint to a Pyrex cap. Four tungsten leads were sealed into the cap, two from the controller to a chromel-alumel thermocouple and two from the VTVM to the specimen. A connector tube in the cap linked the system to an electronic vacuum gage, measuring from 0 to 20 millimeters of mercury. The tube was placed in the split tube furnace shown in Figure 2 and the curing temperature was maintained by the powerstat and controller.

III. RESULTS AND DISCUSSION

A. Rubidium Silicate Cements

The development of a potassium silicate-based strain gage cement was based on the simple conception of ion mobility in a vitreous structure. If the thresholds of dielectric breakdown of a series of alkali and alkaline earth silicates are considered, they are found to conform quite well to the relations between the dimensions of the silicate network and modifier cations. More simply expressed, a small univalent ion can move more easily within a given silicate network than a larger univalent ion or a divalent ion of

similar size.

Thus it appeared feasible that a cement of greater resistance to electrical potential might be achieved simply by taking the next step in increasing the size of the alkali cation, i.e., the replacement of potassium with rubidium.

A rubidium disilicate suspension was prepared by melting rubidium carbonate (supplied by American Potash and Chemical Corporation) and Ottawa special silica in the correct proportion. The chemicals contained in a platinum crucible were fired in a gas pot furnace at 1540°C. The product was dissolved in boiling distilled water to form a clear suspension of about 50 per cent solids by weight, having a specific gravity of 1.62.

In admixtures with silica, fairly high tensile strengths are attained by R-1, as shown in Table I. However, resistances are much lower than desired and a 650°F cure is indicated by the electrical tests. Introduction of a vacuum at 220°F is somewhat effective. Decreasing the disilicate content by one-half (R-2) results in higher resistances and lower strengths; the electrical properties are poorer than those exhibited by potassium silicate cements. Incorporation of Ludox AS, an ammonium stabilized colloidal silica which enhances the resistivity of a aluminum phosphate cements, effects improved electrical behavior in formulation R-3. Although the benefits of a vacuum is again displayed, the indicated curing temperature remains 650°F as it is for R-1 and R-2. This is borne out by a close to linear relationship between resistance and temperature after a 700°F cure.

The cation content (which appears to be the mobile conductor) was decreased further by preparation of rubidium trisilicate. The mode of firing remained the same as for the disilicate. The maximum temperature was 1475°C where complete melting was evident. Maximum solubility of the trisilicate was 17.4 per cent by weight in contrast to the more readily dissolved disilicate.

Formulations containing the trisilicate binder, silica filler and goulac suspending agent form a rather weak bond as recorded in Table II. Electrical properties are comparable to those of the best potassium silicate cements made in the previous program. However, the latter exhibited adequate curing at less than 220°F. A much higher degree of curing at

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Table I. PROPERTIES OF RUBIDIUM DISILICATE CEMENTS

Formulation No.	R-1		R-2		R-3			
Cured Strength (psi)[1]	750A		450A		400A			
Cure °F[2]	220	220 Vacuum	180	300	Room Temp. Vacuum	250 Vacuum	220	700
Resistance[3] (megohms) vs. Temperature (°F)								
200	0.44	28.0	0.0055	0.35	0.018	300	19	0.20
300	0.07	0.33	0.024	4.2	0.18	17	1.3	6.8
400	0.006	0.023	0.135	0.31	0.50	0.75	0.47	39
500	0.005	0.015	0.045	0.042	0.125	0.115	0.09	12
600	0.0031	0.014	0.011	0.011	0.030	0.028	0.028	2.4
700	0.0030	0.009	0.009	0.0085	0.028	0.039	0.020	0.23
800	0.0008	0.0027	0.0047	0.0048	0.026	0.023	0.016	0.033
900	0.0003	0.0008	0.0015	0.0025	0.0062	0.006	0.0035	0.005
Strength (psi) after test	1050A	265A	465A (two heating cycles)	500A	475A	---	675A	715A
Composition	10g Ottawa Microsil SiO$_2$ 4cc 50 per cent Rb$_2$O·2SiO$_2$ 1cc 1 per cent Goulac		10g Ottawa Microsil SiO$_2$ 2cc 50 per cent Rb$_2$O·2SiO$_2$ 3cc 1 per cent Goulac		10g Ottawa Microsil SiO$_2$ 2cc 50 per cent Rb$_2$O·2SiO$_2$ 2cc 1 per cent Goulac 1.5cc Ludox 4S			

(1) Strengths determined after a 220°F cure for approximately one hour.
(2) Curing times were one hour except for R-2 specimens in which the 180°F sample was heated for 45 minutes and the 300°F sample was cured for two hours.
(3) Heating rate for tests: 200-900°F in ca. 8 minutes.

Table II. PROPERTIES OF RUBIDIUM TRISILICATE CEMENTS

Formulation No.		RT-1			RT-2		
Cured Strength (psi)[1]	Cure °F [2]	75A			475A		
		220[3]	220[4]	350[4]	220[3]	220[4]	350[4]
Resistance (megohms) vs. Temperature (°F)	200	32	300	200	750	∞	∞
	300	50	120	160	55	300	200
	400	8	18	22	3.0	11.2	7.2
	500	0.70	0.70	1.2	0.21	0.55	0.25
	600	0.13	0.12	0.75	0.038	0.09	0.064
	700	0.090	0.078	0.08	0.028	0.055	0.055
	800	0.044	0.046	0.055	0.036	0.049	0.034
	900	0.017	0.016	0.090	0.024	0.023	0.011
Strength (psi) after test		0A	225A	0A	25A	150CA	275CA
Composition		10cc Ottawa Microsil SiO$_2$ 5cc 17.4 per cent Rb$_2$O·3 SiO$_2$ 1cc 1 per cent Goulac			5g Ottawa Microsil SiO$_2$ 5cc 17.4 per cent Rb$_2$O·3 SiO$_2$ 1cc 1 per cent Goulac		

(1) Strengths determined after a 220°F cure for one and one-half hour.

(2) Curing times were three hours for a 220°F cure and 30 minutes for a 350°F cure.

Heating rates for tests: (3) 200-900°F in ca. three to three and one-half minutes.

(4) 200-900°F in ca. eight minutes.

220°F is exhibited by the RT (trisilicate) cements as compared to the R compositions. There appears to be no significant increase in resistance after a 350°F cure over a 220°F cure. As in the case of the disilicate, a curing temperature of about 650°F is apparent from the resistance versus temperature experiments illustrated in Figure 6 (see descriptive legend to Figures 6-17, preceding Figure 6). The superior electrical properties of RT-2 cured at 700°F wanes at higher temperatures.

B. Phosphate-Bonded Cements

1. Previous Work

In the previous phases of the high temperature cement program, compositions were based mainly on the monoaluminum dihydrogen phosphate binder. A quite pure source of this material was obtained as a fifty per cent suspension from the Victor Chemical Company. It was found that this binder used in conjunction with an inert filler required a high curing temperature (ca. 750°F) and thermal cycling and adherence properties were poor. For these reasons, an additive study was made.

Chromic acid addition improved adhesion properties markedly and contributed to a reduction in curing temperature. The use of finely divided silica for the filler was almost mandatory for adequate thermal cycling properties. Other additives were found that improved properties in one or more respects, the most effective being hydrated alumina.

A composition was developed which became the basis for the present work and was formulated as follows:

    100 grams micronized silica
      3 grams hydrated alumina
      5 grams chromic acid anhydride
     35 cc monoaluminum dihydrogen phosphate (50%)
     20 cc distilled water containing a suspending agent.

This composition permitted effective curing at 400°F and also displayed high strength. However, the actual temperature for full curing of this material is about 630°F.

It was anticipated that continued additive studies would bring about further lowering of the required curing temperature of the aluminum phosphate system.

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Differential thermal analyses (DTA) of compositions containing phosphoric acid as the binder indicated that quite low temperature curing might be accomplished in a vacuum. Although vacuum curing is not highly practical for strain gage installation, it was hoped that it would be useful in significantly lowering temperature requirements.

2. Phosphoric Acid Cements

Although previous work has shown that the monoaluminum dihydrogen phosphate binder will normally be cured at temperatures well below that of phosphoric acid, the DTA studies indicated that the latter would be more amenable to low temperature vacuum curing. The acid bonded compositions were based on information gained in investigations of the aluminum compound. Chromic acid was incorporated as before, as a corrosion inhibitor and adhesion promoter. Chrome oxide (Cr_2O_3) was also included. The hydrated alumina content was increased to develop alumino-phosphate bonding. Silica was still required as the filler. Alumina imparted inferior thermal cycling properties and zircon, although providing very high tensile strengths, was more electrically conductive.

It was found that although vacuum treatment could reduce time of curing and increase the electrical resistance at a specific curing temperature, it did not reduce the actual temperature requirement. The best compositions required at least 400°F and were hygroscopic on cooling. Much higher temperatures are required to eliminate the moisture sensitivity of the phosphoric acid systems.

The vacuum system employed was operated at 80 to 100 microns of mercury pressure. Higher vacuums were considered totally impractical. However, to fully evaluate the vacuum technique, a phosphoric acid composition was subjected to conditions of 5 microns at 250°F. Its behavior on testing was similar to a 250°F treatment at atmospheric pressure. Another specimen was placed in a chamber at room temperature and a pressure of 10^{-6} millimeters of mercury. This also was ineffective.

In view of the failure of vacuum curing and the inherently high curing temperature of the phosphoric acid binder, this system was dropped in favor of the investigation of aluminum phosphate.

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3. **Commercial Cements**

Various experiments were conducted with three commercial cements: Allen P-1 and PBX obtained from the Baldwin-Lima-Hamilton Corporation, and "H" Cement supplied by William T. Bean, for purposes of comparison with cements developed at ARF. The BLH Corporation cements are available in kit form, i.e., liquid and powder in separate bottles, and "H" in ready mix form.

Tabulation of the data in Table III reveals that no clear superiority in all properties is displayed by any one cement over the other two. Allen P-1 shows the best curing at 220°F; however, it lags behind PBX and "H" electrically after a 350°F cure and is also the weakest cement. "H" cement displays the highest resistances after a 350°F cure, a leadership which it retains up to about 1200°F. Whereas both PBX and "H" show a marked decrease in conductivity when pretreated at 350°F, Allen P-1 undergoes no significant improvement. All cements display the characteristic dip in resistance (400° - 450°F for PBX and "H", and 600°F for Allen P-1) which signalizes inadequate curing. PBX and "H" possess similar strengths. Properties of "H" cement, which has perhaps the best combination of properties, are illustrated in Figure 7.

4. **Monoaluminum Dihydrogen Phosphate Cements-Additive Study**

The additive study for the aluminum dihydrogen phosphate cements was based in part on the article by W. D. Kingery, "Fundamental Study of Phosphate Bonding in Refractory Bodies," J. Am. Cer. Soc., Vol. 33, pp. 239-247, 1950. This paper was concerned mainly with additions to phosphoric acid, and previous compositional studies disclosed that materials reactive with the acid might effectively combine with free phosphate upon heating of the aluminum compound, thus effecting a cure without need of volatilizing released phosphoric anhydride.

A satisfactory formulation described earlier, B52, developed from earlier investigations has the specific composition:

10g	Ottowa special silica
0.5g	Chromic anhydride
0.3g	Alumina trihydrate (Alcoa C-31)

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Table III. PROPERTIES OF MONOALUMINUM DIHYDROGEN PHOSPHATE CEMENTS

Formulation[1]	Alumina[2] (g)	Additives (g)	Strength[3] As Cured (psi)	220°F Cure[4] 200	220°F Cure[4] Minimum/Temp.	220°F Cure[4] 900	220°F Cure[4] Strength[3] (psi)	350°F Cure[5] 200	350°F Cure[5] Minimum/Temp.	350°F Cure[5] 900	350°F Cure[5] 1200	350°F Cure[5] 1600	350°F Cure[5] Strength (psi)
Standards:													
76	0.3g C-35	None	800C	(a) 0.10 / (b) 0.09	0.0028/375 / 0.0034/350	0.21 / 0.175	(a) 25A / (b) 1000C	(a) ∞	0.13/500	0.31	0.12	0.013	700A
85	0.3g MA	None	750A	(a) 0.09 / (b) 1.0	0.0024/350 / 0.007/350	0.73 / 0.75	(a) 0A / (b) 560A	(a) ∞	0.20/500	0.53	0.15	0.010	600A
Commercial Cements:													
Allen P-1			350A	(a) 200 / (b) 500	0.195/575 / 0.22/575	0.47 / 0.40	(a) 125C / (b) 600C	(a) ∞ / (b) 750	0.50/600 / 0.195/600	0.39 / 0.38	0.078	0.0018	225CA / 125CA
H Cement			1075CA	(a) 12 / (b) 50	0.011/375 / 0.24/425	0.70 / 0.32	(a) 250CA / (b) 1125CA	(a) ∞ / (b) 500; (b) 750	None / 2.2/500	1.0 / 1.1	0.080	0.005	600CA / 900CA
PBX			1375CA	(a) 2.5 / (b) 8.5	0.045/450 / 0.060/400	0.43 / 0.40	(a) 600CA / (b) 1075C	(a) 750 / (b) 750	1.4/500 / 1.0/525	0.13 / 0.70	0.043	0.006	400A / 150C
Miscellaneous Additives:													
84	0.3 MA	0.4 CaO	1000C	(a) 1.0 / (b) 3.9	0.017/375 / 0.019/400	0.77 / 0.18	(a) 150A / (b) 825A	(a) ∞	0.65/500	0.90	0.27	0.006	500A
86	0.3 MA	0.5 SnO	150C	(a) 0.0125	0.0026/400	0.0044	(a) 350A / (b) 1450A	(a) 0.0075	0.0044/500	0.0020	(200°-900°F only)	0.016	1275A
87	0.3 MA	0.5 SnO₂	---	(a) <1.0 / (b) 6.4	0.015/400 / 0.013/400	1.2 / 1.0	(a) 100A / (b) 1450A	(a) 400	0.40/500	0.53	0.145	0.0052	950A
116	0.3 MA	0.5 CaF₂	1250CA	(a) 0.80 / (b) 5.2	0.006/400 / 0.023/350	1.6 / 3.0	(a) 475CA / (b) 750CA	(a) 750	0.36/475	1.8	0.41	0.085	300CA
121	0.3 MA	0.6 La(NO₃)₃ · 6 H₂O	450A	(a) 1.0 / (b) 14	0.0055/375 / 0.023/375	2.1 / 4.8	(a) 125A / (b) 450CA	(a) ∞	6.5/600	4.5	1.1	0.0017	125A
Zinc Compounds:													
77	0.5 C-35	0.5 Zn(C₂H₃O₂)₂ · 2 H₂O	1100A	(b) 0.80	0.0017/450	0.37	750A	(No Tests)	1.0/500	0.24	0.125	0.0027	825A
78	0.5 C-35	0.3 ZnO	1100A	(b) 8.0	0.028/400	0.22	400A	(a) ∞	2.8/900	3.2	(200°-900°F only)	0.0034	475A
79	0.5 C-35 / 0.7 MA	0.3 ZnO	---	(b) 45	0.0044/400	1.5	425A	(a) ∞					
80	0.8 MA	0.3 ZnO, 0.5cc G	940A	(b) 35	0.010/400	1.7	300A	(a) ∞	1.2/550	0.75	0.09	0.0048	550A
81	0.7 MA	0.6 ZnO, 0.7cc G	150A	(a) 150 / (b) 200	0.18/400 / 0.17/400	1.3 / 3.8	(a) 50A / (b) ---	(a) ∞	3.0/500	2.5	0.36	0.0031	100A
82	0.3 MA	0.3 ZnO	1125CA	(a)* 6.0 / (b)* 49	0.022/425 / 0.044/400	0.60 / 0.70	(a) 0A / (b) 775C	(a) ∞	4.5/500	1.3	0.32		475A
83	0.3 C-35	0.5 ZnO	625A	(a) 3.5 / (b) 12.5	0.0009/375 / 0.0015/350	0.06 / 0.054	(a) 125A / (b) 775C	(a) ∞	None	0.105	0.07		200A
88	0.2 MA	0.2 ZnO	450A	(a) 1.9 / (b) 9.0	0.0026/490 / 0.009/400	1.6 / 1.0	(a) 150C / (b) 425A	(a) ∞	0.42/450	0.60	0.26	0.026	140A
102	0.6 Al(C₂H₃O₂)₃	0.3 ZnO	875C	(a) 4.4 / (b) 60	0.032/400 / 0.132/400	0.90 / 0.80	(a) 350C / (b) 900CA	(a) 750	0.80/500	0.22	0.078	0.0018	950CA

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Table III PROPERTIES OF MONOALUMINUM DIHYDROGEN PHOSPHATE CEMENTS (cont.)

Formulation[1]	Alumina[2] (g)	Additives (g)	Strength As Cured (psi)	220°F Cure[4] 200	Minimum/Temp.	900	Strength[3] (psi)	350°F Cure[5] 200	Minimum/Temp.	900	1200	1600	Strength (psi)
Magnesium Compounds													
92	0.3 MA	0.2 MgO	675CA	(a) 4.5 (b) 34	0.010/375 0.065/400	1.9 3.6	0C 250CA	(a) 500	1.5/450	2.0	0.50	0.044	375CA
93	0.3 MA	0.2 MgO(1500°C/15 hr.)	800C	(a) 12 (b) 26	0.09/400 0.05/400	2.3 2.2	0A 250A	(a) 750	1.6/500	2.6	0.30	0.012	275A
95	0.3 MA	0.4 MgO(1500°C/15 hr.)	500A	(a) 8 (b) 500	0.118/375 0.70/400	1.0 2.5	75C 200A	(a) 750	3.0/450	3.3	0.72	0.017	100A
101	0.6 Al(C₂H₃O₂)₃	0.2 MgO(1500°C/15 hr.)	625C	(a) 500 (b) 750	3/525 1.5/525	2.0 0.70	200C 550C	(a) ∞	2.0/550	1.5	0.21	0.012	400C
100	0.3 MA	1.0 Mg(C₂H₃O₂)₂·4H₂O	. .	(a) 9.2	0.165/425	1.9	0C	(a) 300	3/450	4.0	0.62	0.017	75C
103	0.3 MA	0.6 MgSO₄	525C	(a) 7.8 (b) 500	0.059/425 0.32/550	1.2 0.65	75C 300C	(a) 50	0.17/475	0.71	0.26	0.010	150C
104	0.3 MA	0.5 Mg₃(PO₄)₂	300C	(a) 5.5 (b) 25	0.042/400 0.125/400	2.1 2.0	50C 125C	(a) 150	0.60/475	1.8	0.62	0.045	50C
113	0.3 MA	2.0 Mg(NO₃)₂·6H₂O	200CA	(a) 100 (b) 90	1.0/375 3.2/375	11.5 4.0	0C 75CA	(a) ∞	12/475	3.1	0.24	0.0048	0C
127	0.3 MA	1.0 MgCl₂·6H₂O	75C	(a) 70 (b) 200	2.6/375 2.6/375	2.2 13.5	0C 50C	(a) ∞	31/500	17.0	1.2	0.010	0A
135	0.3 MA	0.4 MgF₂	625C	(a) 1.0	0.004/400	2.9	550CA	(a) 750 (b) 200	0.27/475 0.28/475	1.7 6.2	0.50	0.007	375CA 50CA
137	0.3 MA	0.6 MgF₂	500CA	(no 110°F cure tests)				(a) 750 (b) 400	0.90/500 0.70/475	3.3 4.2	0.50	0.002	500CA 150CA
115	0.3 MA	0.5 MgF₂, 0.5cc HNO₃	400A	(a) 28 (b) 140	0.115/375 0.60/375	2.7 5.0	175A 200A	(b) ∞	3.0/475	6.9	0.65	0.0020	100A
134	0.6 MA	0.4 MgF₂, 1cc G	850CA	(a) 2.0	0.025/350	4.8	225CA	(a) 750 (b) 100	1.2/475 0.19/475	5.0 2.4	0.62	0.003	425CA 525CA
138	0.4 MA	0.4 MgF₂	700CA	(a) 45 (b) 150	0.048/425 0.21/400	7.7 9.0	225CA 225C	(a) 500 (b) 500	0.50/475 0.31/475	2.0 5.5	1.0	0.004	125CA 600C
112	0.6 Al(C₂H₃O₂)₃	0.4 MgF₂	775C	(a) 7.5 (b) 55	0.012/400 0.031/425	1.6 2.0	150C 600C	(a) ∞	1.0/500	2.1	0.35	0.006	600C
114	1.2 Al(NO₃)₃·9H₂O	0.4 MgF₂	250CA	(a) 3.0 (b) 200	0.008/350 1.3/375	3.4 4.8	125CA 75CA	(a) ∞	10.5/475	6.3	0.36	0.0045	125CA
131	0.3 MA	0.5 MgF₂, 1.0 MgCl₂·6H₂O	100C	(a) 45 (b) 200	2.0/350 5.0/350	20 19.5	50C 50C	(a) ∞	37/475	30	1.2	0.0004	0CA
133	0.3 MA	0.6 MgF₂, 1.0 Mg(NO₃)₂·6H₂O	350A	(a) 55 (b) 160	0.22/375 0.50/375	4.8 3.6	75A 325A	(a) 300 (b) 300	4.6/475 1.6/475	2.0 0.80	0.60	0.0065	75A 100A
106	0.3 MA	0.6 Mg Al₂O₄	675C	(b) 110	0.042/425 0.24/425	6.2 6.1	600CA 650CA	(a) 750	3.9/500	9.5	1.2	0.006	500CA

Table III: PROPERTIES OF MONOALUMINUM DIHYDROGEN PHOSPHATE CEMENTS (cont.)

RESISTANCE (megohms) VS. TEMPERATURE [7]

Formulation[1]	Alumina[2] (g)	Additive (g)	Strength[3] As Cured (psi)	230°F Cure[4] 200	230°F Min./Temp.	230°F 900	230°F Strength[5] (psi)	180°F Cure[5] 200	180°F Min./Temp.	180°F 900	1200	1600	Strength (psi)
Magnesium Compounds (cont.):													
107	0.6 Al(C₂H₃O₂)₃	0.6 Mg Al₂O₄	600C	(a) 40 / (b) 40	0.19/425 / 0.28/450	2.9 / 2.2	75C / 75OC	(a) —	2.5/475	3.8	0.40	0.022	275C
108	None	1.0 Mg Al₂O₄	750CA	(a) 26 / (b) 52	0.042/425 / 0.082/425	4.1 / 4.0	150CA / 750CA	(a) 750	2.3/475	6.2	0.90	0.022	250CA
109	None	0.5 Mg Al₂O₄	1225CA	(a) 11.0 / (b) 17.5	0.050/425 / 0.155/450	3.1 / 2.1	125A / 700C	(a) 200⁺	0.50/500	2.0	0.62	0.039	190CA
136	0.4 MA	0.4 Mg Al₂O₄	1675A	(a) 9.0	0.0036/425	5.0	450C	(a) 750 / (b) 300	0.80/500 / 0.70/475	2.0 / 4.5	0.36	0.006	125CA / 75CA
140	0.6 MA	0.3 Mg Al₂O₄	600CA	(a) 5.0	0.021/400	8.2	250CA	(a) 750 / (b) 500	1.2/500 / 0.95/475	6.5 / 8.0	1.0	0.004	300CA / 200C
Yttrium Compounds (6):													
88	0.3 MA	0.4 "M" Y₂O₃	900C	(a) 11 / (b) 30	0.23/350 / 0.26/350	0.85 / 1.2	150C / 800C	(a) 200	1.1/500	<1	0.13	0.004	400C
90	0.3 MA	0.4 "M" Y₂O₃(1100°C/4 hr.)	500C	(a) 65 / (b) 200	0.60/300 / 1.6/450	2.8 / 1.9	0C / 975C	(a) 500	1.0/500	2.0	0.50	0.030	625C
91	0.3 MA	0.4 "M" Y₂O₃(1500°C/15 hr.)	1050C	(a) 0.40 / (b) 2.2	0.012/375 / 0.030/325	0.47 / 2.5	0C / 675C	(a) 750	0.45/450	1.8	0.62	0.042	450C
94	0.3 MA	0.4 Y₂O₃	500A	(a) 3.4 / (b) 30	0.030/375 / 0.13/375	2.4 / 4.0	125A / 645A	(a) 200	1.0/500	5.7	0.95	0.052	275A
96	None	0.3 "M" Y₂O₃(1100°C/4 hr.)	375A	(a) 2.0 / (b) 500	0.012/400 / 0.185/450	1.1 / 1.1	75A / 150A	(a) 750	0.32/450	2.1	1.0	0.097	150A
105	0.6 Al(C₂H₃O₂)₃	0.4 Y₂O₃	550C	(a) 10 / (b) 80	0.097/400 / 0.31/450	2.1 / 0.90	75C / 425C	(a) 750	4.7/500	2.1	0.30	0.008	125C
125	0.3 C-35	0.4 Y₂O₃	1050CA	(a) 4 / (b) 17	0.011/400 / 0.10/400	2.0 / 2.3	250CA / 950CA	(a) —	0.70/500	2.5	0.90	0.042	400CA
120	0.3 MA	0.6 Y₂(C₂O₄)₃·9H₂O	375C	(a) 5.0 / (b) 5.7	0.063/350 / 0.090/325	6.0 / 1.6	125CA / 250CA	(a) 750	1.6/500	4.3	1.1	0.096	225CA
99	0.6 Al(C₂H₃O₂)₃	0.3 Y₂(C₂O₄)₃·9H₂O	425C	(a) 28 / (b) 72	0.32/375 / 0.12/375	4.9 / 5.2	0A / 475CA	(a) 750	4.0/500	3.2	0.39	0.035	375A
145	0.3 MA	0.4 Y₂(C₂O₄)₃·9H₂O	600A	(a) 3.8 / (b) 10.5	0.029/350 / 0.090/350	4.2 / 7.8	375CA / 375CA	(a) 300 / (b) 300	0.95/500 / 0.60/375	4.4 / 4.5	1.5	0.125	280C / 100CA
123	0.3 C-35	1.0 Y(NO₃)₃·6H₂O	625CA	(a) 10 / (b) 200⁺	0.12/375 / 0.36/375	7.1 / 3.9	0A / 50CA	(a) 750	1.6/500	2.7	0.80	0.060	125A
124	1.2 Al(NO₃)₃·9H₂O	1.0 Y(NO₃)₃·6H₂O	250A	(a) 90 / (b) 150	0.022/375 / 0.035/375	0.50 / 2.9	0A / 0A	(a) —	3.0/475	0.90	0.095	0.0062	25CA
119	0.3 MA	0.3 Y(NO₃)₃·6H₂O	825A	(a) 3 / (b) 10.0	0.0036/425 / 0.030/375	0.68 / 2.3	325CA / 875CA	(a) 750	1.5/475	5.1	1.0	0.070	750CA

Table III. PROPERTIES OF MONOALUMINUM DIHYDROGEN PHOSPHATE CEMENTS (cont.)

Formulation[1]	Alumina[2] (g)	Additives (g)	Strength[3] As Cured (psi)	RESISTANCE (megohms) VS. TEMPERATURE (°F)										
				220°F Cure[4]				350°F Cure[5]						
				200	Minimum/Temp.	900	Strength[3] (psi)	200	Minimum/Temp.	900	1200	1600	Strength (psi)
Yttrium Compounds (cont.)													
144	0.3 MA	0.4 Y(NO3)3 · 6H2O	600A	(a) 6.3 (b) 12.0	0.027/375 0.022/425	3.4 4.1	75CA 675CA	(a) 750 (b) 500	6.2/475 1.3/450	8.0 7.9	1.8	0.115	175A 150CA
147	0.3 MA	0.5 Y(NO3)3 · 6H2O	1175A	(a) 9.0 (b) 15.0	0.042/375 0.030/375	1.7 5.5	225A 575CA	(a) 750 (b) 750	1.5/500 2.1/475	2.5 10.0	0.46	0.026	475A 150CA
98	0.3 MA	0.6 Y(NO3)3 · 6H2O	350A	(a) 20	0.40/400	7.4	50C	(a) 130	0.8/475	4.6	0.70	0.038	150C
132	0.3 MA	0.8 Y(NO3)3 · 6H2O		(a) 7 (b) 39	0.068/325 0.235/375	6.3 9.4	200CA	(a) 750	8.5/475	11.2	1.3	0.056	50A
118	0.3 MA	1.0 Y(NO3)3 · 6H2O	250A	(a) 16 (b) 200	0.25/375 1.8/375	2.9 12.4	75CA 50CA	(a) ∞	9.2/525	7.8	1.1	0.072	200CA
130	0.3 MA	1.0 Y(NO3)3 · 6H2O,0.5cc AP	450CA	(a) 5.2	0.049/350	13.7	50CA	(a) 200	1.3/475	2.3	0.90	0.067	0A[6] 125SCA
126	0.3 MA	1.5 Y(NO3)3 · 6H2O	350CA	(a) 28 (b) 200*	0.16/375 1.1/375	6.7 9.5	0CA[7] 50CA[7]	(a) ∞	6.2/475	7.9	0.62	0.034	125SCA
148	0.5 MA	0.4 Y(NO3)3 · 6H2O	875A	(a) 6.0 (b) 31	0.0135/400 0.20/375	5.4 6.7	100CA 300A	(a) 400 (b) 250	6.1/475 1.9/450	5.2 2.4	0.80	0.045	250A 50A
Combinations of Magnesium and Yttrium Compounds													
111	None	0.6 MgAl2O4, 0.4 Y3(C2O4)3 · 9H2O	475CA	(a) 10.0 (b) 70	0.26/425 0.52/425	4.3 6.6	50CA 225CA	(a) 200	2.2/500	5.0	1.0	0.225	50C
128	0.3 MA	1.0 Mg(NO3)2 · 6H2O, 0.6 Y(NO3)3 · 6H2O	150CA	(a) 30	0.090/375	3.3	0C	(a) ∞	39/475	10.5	0.75	0.034	0CA[8]
142	0.3 MA	0.5 MgCl2 · 6H2O, 0.3 Y(NO3)3 · 6H2O	0C	(a) 90 (b) 300	3.6/375 3.0/375	30 20	0C 0C	(a) 750 (b) 500	22.475 9.0/475	74 26.5	4.3	0.065	0C 0C
129	0.3 MA	0.4 MgF2, 0.4 Y(NO3)3 · 6H2O	400A	(a) 3.8 (b) 110	0.022/375 1.5/400	14 9.7	50CA 125A	(a) ∞	10.5/475	13.5	0.90	0.015	125A
143	0.3 MA	0.3 MgF2, 0.3 Y(NO3)3 · 6H2O	500C	(a) 1.0 (b) 7.5	0.0105/375 0.045/375	2.1 2.1	375CA 300CA	(a) 750 (b) 250	3.4/475 1.1/450	3.6 4.5	0.70	0.058	525CA 125C
141	0.3 MA	0.3 MgAl2O4, 0.3 Y(NO3)3 · 6H2O	875CA	(a) 3.8 (b) 11.5	0.0052/425 0.033/350	4.5 7.0	225CA 550CA	(a) 750 (b) 400	4.0/475 1.5/450	7.5 13.0	1.1	0.050	325CA 75CA
149	0.3 MA	0.4 MgAl2O4, 0.2 Y(NO3)3 · 6H2O	1250A	(a) 5.2 (b) 15	0.0135/375 0.042/375	4.4 3.6	150CA 650A	(a) 750 (b) 400	3.8/475 0.90/450	6.2 6.3	0.90	0.038	250A 350CA

(1) Formulations contain the basic ingredients: 10 grams silica (Ottawa Special Microsil), 0.5 g chromic anhydride, 3.5 cc 50 per cent monoaluminum dihydrogen phosphate, and 2 cc 1 per cent guaiac suspension, plus chemicals denoted under the columns "alumina" and "additives."

(2) C-35 is Al2O3 3H2O marketed by Alcoa. MA is Gallon "Alucer" MA, a highly pure active form of "gamma" Al2O3.

(3) As-cured strength was determined after a 220°F cure. The suffixes C and A indicate the type of failure, cohesive and adhesive. Strength tests were conducted after the samples had cooled to handling temperature.

(4) Two heating rates were employed for 200°-900°F tests of samples cured for three to four hours at 110°C. Samples cured overnight are specified with a superscript; (a)* or (b)*. A rapid rate is that in which 900°F was attained in three to three and one-half minutes is denoted as (a); a moderate rate of about eight to nine and one-half minutes is denoted as (b).

(5) Samples were cured at 350°F for one hour prior to testing, except for B88, B90, B91, B94, and B96, which were cured for 15 minutes. A moderate heating rate of 200°-1600°F in about 26 to 30 minutes is denoted (a); a fast rate of 200°-900°F in three to three and one-half minutes is denoted (b).

(6) "M" Y2O3 denotes Michigan Chemical Titria. The unmarked Y2O3 and the oxalate and nitrate were obtained from Lindsay Chemical Company.

(7) Samples were cycled to 900°F and cooled twice prior to strength determination.

(8) Samples were cycled to 1600°F and cooled twice prior to strength determination.

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3.5cc   Monoaluminum dihydrogen phosphate (50%)

2.0cc   Goulac suspending agent solution (1%)

A similar formula, B76, contains a purer hydrate, C-35, in place of C-31; it exhibits properties similar to B52. Substitution of a highly pure, finely divided, active "gamma" alumina, Gulton "Alucer" MA, for C-31, results in higher electrical resistances at elevated temperatures. Properties of this cement (B85) appear in Table III and are graphically presented in Figure 8. Various compounds were added to the basic composition, B85, in an effort to lower curing temperatures and improve electrical properties while retaining strength. The additives are broken down into five groups: miscellaneous additives, zinc compounds, magnesium compounds, yttrium compounds, and combinations of magnesium and yttrium compounds. The properties of all cements are listed in Table III.

a.   Miscellaneous Additives

Grouped under this heading are additives which for various reasons did not merit extensive study. The incorporation of CdO (B84), SnO_2 (B87), or CaF_2 (B116) does not improve the electrical properties of B85, although good strengths are exhibited by these cements. Use of SnO (B86) actually increases the conductivity of the composition.

On the other hand, $La(NO_3)_3 \cdot 6H_2O$ considerably enhances good electrical properties (B121). After a 350°F cure for one hour, resistances are higher than one megohm up to 1200°F. A 220°F cure is inadequate as illustrated by the minimum resistances at 375°F. Strengths are moderately good. However, the La^{+3} containing cement suffers from a rather short jar life; setting occurs within five hours. Thus, it would be limited to preparation from a kit form and use in about three hours.

b.   Zinc Compounds

The addition of zinc oxide to the B76 type of composition revealed possibilities of a 350°F cure (B78 and B79). Increasing the ratio of ZnO to C-35 (B83) has a deleterious effect on electrical properties. The use of zinc acetate in place of zinc oxide (B77) yields a cement of good strength but poor electrical resistance.

Substitution of Gulton "Alucer" MA for the C-35 aluminum trihydrate significantly improved electrical properties, especially at the higher

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temperatures. The composition which appears to possess the most satisfactory combination of properties is B82, illustrated in Figure 9. An excess of MA and/or ZnO weakens the cement without an accompanying increase in resistance; there are indications, however, that a better degree of curing is achieved at a lower temperature with additional pure alumina.

Incorporation of aluminum acetate in place of MA (B102) yields a mixture which cures adequately and exhibits good strengths. Poor electrical properties at elevated temperatures precludes its acceptability. Lumping of zinc oxide in the various formulations was prevented by ball milling the solid constituents prior to liquid addition.

C. Magnesium Compounds

The use of magnesium oxide results in cements (B92, B93, and B95) similar electrically (as depicted in Figure 10) and slightly inferior in strength to Zn^{+2} containing B82. A definite drawback of these cements is their extremely short shelf life of less than two hours. A forecast of this short setting time was observed in the generation of heat during preparation of the cement. Attempts to render the MgO inert by calcining at 1100°C and 1500°C were unsuccessful. A composition (B101) utilizing aluminum acetate instead of MA cures well at 220°F and shows good electrical properties. Very poor shelf life is also the detracting factor for B101.

To retain the good electrical properties of cements containing Mg^{+2} while extending shelf life, more inert forms of magnesium compounds were investigated. The sulfate (B103) and phosphate (B104) impart good shelf life but causes deterioration in strength and electrical properties. The setting reaction of MgO is also obviated by substitution of the acetate (B100), nitrate (B113) and chloride (B127). Excellent electrical behavior is demonstrated by these cements, particularly by B113 and B127 after a 220°F cure. Figure 11 illustrates the resistance-temperature relationship for B127. Unfortunately these favorable qualities are annulled by extreme fragility of these formulations containing the hydrates.

Experiments with compositions employing MgF_2 as an additive suggested a more extensive study of their moderately good all around properties. Increased amounts of the fluoride has a beneficial effect on curing properties, as does a small amount of nitric acid, but both weaken the cement slightly. B134, which is typical of MgF_2 cements, is presented in Figure 12.

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An undesirable characteristic of B135, B137, B115, B134, and B138 is what may be termed poor recovery when cycled to 1600°F; on cooling, the resistance versus temperature curve is somewhat below the heating curve.

Substitution of aluminum acetate for MA (B112) results in a cement of fairly good strengths and curing; however, a decrease in resistance at higher temperatures is evident, as it is for B114 which incorporates aluminum nitrate. B114 further suffers from poorer physical properties. No advantages could be observed by replacement of Gulton "Alucer" MA by either of the aluminum compounds.

Investigations were conducted in the magnesium systems to incorporate the favorable properties of two additives. Thus the fluoride was added to formulations in combination with both the chloride (B131) and the nitrate (B133). Good electrical properties are exhibited by these cements, but strengths are only slightly improved over compositions B113 and B127 which do not include the fluoride. Furthermore "poor recovery" in resistances exists after a 1600°F cycle. The behavior shown in Figure 11 is typical of the properties of these double additive mixtures.

A 50 mol per cent magnesia-alumina spinel which is quite stable chemically, appeared attractive as a means of introducing Mg^{+2} in an inert form. Properties similar to those of B134 containing MgF_2 are displayed by a spinel cement, B106, graphically presented in Figure 13. A slight deterioration in electrical properties at elevated temperatures was the only change when aluminum acetate was substituted for MA in B107. Complete removal of alumina from the spinel formulation (depending upon the alumina in spinel to perform the same task) causes slight reduction of the observed electrical resistances on heating (B108, B109). Increasing the MA content results in slight improvement of high temperature resistance. Inability on cooling from 1600°C to attain resistances shown on the heating cycle typifies the spinel group.

d.   Yttrium Compounds

Excellent electrical resistances are exhibited by yttria containing cements, not only on heating but also on cooling. This good recovery after a 1600°C cycle contrasts nicely with the magnesium cements. Strengths are comparable with those of magnesium formulations. However, use of

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yttria as an additive is handicapped by the same impedance as for magnesia, i.e., extremely short shelf life. Calcination of yttria to minimize its activity does not noticeably prolong the shelf life, but does yield higher resistances at elevated temperatures. A very pure form of yttria (99.99) obtained from Lindsay Chemical Company produces a cement with electrical properties similar to those of the high-fired, less pure Michigan Chemical oxide, but with an equally short jar life. Resistance-temperature curves for this cement appear in Figure 14.

Replacement of MA alumina by either aluminum acetate (105) or C-35 (B125) is not beneficial; slight increases in conductivity result in both instances.

As in the case of magnesium studies, investigations of other components of yttrium was suggested by the early experiments. Addition of yttrium oxalate to the basic composition, B85, yields a cement (B120) of moderate strength which cures quite well at 350°F and exhibits good electrical resistances up to 1600°F (see Figure 15). A very slight increase in strength and mild decline in degree of curing results from decreasing the oxalate content (B145). The high temperature deterioration in electrical properties with replacement of MA by aluminum acetate occurs for yttrium oxalate (B99) as it does for some of the other additives.

Utilization of the oxalate of yttrium in place of the oxide extends shelf life considerably although not indefinitely. A very slow thickening in the consistency of the cement can be noticed after about three hours. This lessening of fluidity continues with time and about 22 hours after formulation the cement in the jar appears to have congealed completely. However, a very thick paste can be formed by mixing. Equilibrium in the setting apparently is not achieved until ultimate gelling occurs after about 40 hours.

A second yttrium salt, the nitrate, was investigated as a high temperature cement additive. Varying amounts ranging from 0.3 gms (B119) to 1.5 grams (B126) were added to the B85 composition. Excellent electrical properties, effective curing at 350°F, and moderate strengths are exhibited by B144 as shown in Figure 16, which employs 0.4 g $Y(NO_3)_3 \cdot 6H_2O$. A lesser amount provides higher strengths but slightly lower resistances; increasing quantities improve electrical properties but weaken the cements. Properties in general compare favorably with those of oxalate

formulations.

The initial behavior of nitrate compositions in the jar is similar to that of oxalate cements, i.e., a gradual thickening is observed. However, after about six hours, the increase in viscosity is arrested for nitrate formulations followed by a thinning until the original consistency is regained.

Substitution of either C-35 aluminum trihydrate (B123) or aluminum nitrate (B124) for MA alumina has no beneficial effect on the cement. Use of the nitrate results in deterioration of electrical properties as well as strength. A slight increase in the MA content (B148) does not significantly alter the properties.

e. Combinations of Yttrium and Magnesium Compounds

Formulations containing a magnesium additive in addition to yttrium nitrate were investigated. Use of either magnesium nitrate (B128) or magnesium chloride (B142) results in cements which are extremely weak. Incorporation of MgF_2 yields compositions (B129 and B143) which exhibit properties similar to those containing $Y(NO_3)_3 \cdot 6H_2O$ alone with perhaps a slight improvement in strength. A shelf life similar to that of yttrium oxalate (B120), i.e., two to three days, limits their value as ready mixes. This moderate jar life is also displayed by a spinel-yttrium oxalate mix (B111) which otherwise displays good all around properties.

The best combinational additive cements were those containing spinel and yttrium nitrate (B141 and B149). Properties of B141 are depicted in Figure 17. Slightly additional strength is contributed by spinel along with retention of good curing at 350°F and excellent electrical behavior.

f. Aging Study

Experiments were conducted to determine the effect of aging on resistance versus temperature. Comparisons were made between commercial "H" cement and an yttrium nitrate composition (B147). Table IV shows the deleterious effect of ambient conditions on the electrical properties of both cements previously cured at 350°F. A 220°F soak for one hour prior to testing partially restores high electrical resistances; however, the values are lower than those obtainable on testing immediately after a 350°F cure. The electrical superiority of B147 is evident from the data. Full restoration

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Table IV. ALUMINUM PHOSPHATE CEMENTS: AGING EFFECTS
ON RESISTANCE (megohms) VS. TEMPERATURE

History:	A[1]		B[2]		C[3]
Formulation:	"H" Cement	B147	"H" Cement	B147	B147
200°F	0.16	0.19	200[+]	70	65
250°F	0.175	0.28	50	13	25
300°F	0.17	0.30	8.0	2.2	11.5
350°F	0.080	0.25	0.90	0.50	6.1
400°F	0.076	0.195	0.35	0.20	3.2
450°F	0.125	0.23	0.21	0.13	1.3
500°F	0.38	0.43	0.50	0.33	1.3
550°F	0.65	0.80	1.0	0.80	1.9
600°F	0.65	1.4	0.80	1.0	5.0
650°F	0.45	3.5	0.35	1.3	6.3
700°F	0.29	3.8	0.18	1.2	6.9
750°F	0.22	3.7	0.125	1.1	7.2
800°F	0.18	3.5	0.10	1.1	7.0
850°F	0.18	3.1	0.105	1.1	5.7
900°F	0.30	2.9	0.32	1.1	4.8
Strength after test	625C	125A	700C	1150CA	150A

(1) A: 220°F/1 hour; 350°F/1 hour; room temperature/16 hours.

(2) B: 220°F/1 hour; 350°F/1 hour; room temperature/ 16 hours, 220°F/1 hr.

(3) C: 220°F/1 hour, 350°F/1 hour, room temperature/16 hours, 350°F/1 hr.

Note: Tests were conducted at a rapid heating rate of 200°-900°F in three to
three and one-half minutes.

of electrical behavior apparently occurs when the cement is subjected to an additional hour at 350°F (see C in Table IV).

Resistance-temperature data were obtained for the cements after they had been exposed to ambient conditions for over 60 hours and then cured at 350°F. Good electrical properties are exhibited by both cements as shown in Table V. Although slight decreases in high temperature resistances occur for B147, the inferiority of "H" cement at these elevated temperatures is still in evidence. Apparently actual testing with B147 must occur very shortly after a 350°F cure to fully exploit its good properties.

g. Rapid Heating Test

In order to more closely simulate the very rapid heating rates encountered in actual practice with strain gage cements, a test was designed incorporating the use of an oxy-gas torch as the source of heat. The cement was applied to one face of an Inconel piece 1/2 inch x 1/2 inch x 1/8 inch and dried. An alumel-chromel thermocouple was imbedded in this same face by covering with a second coat. A chromel lead was silver soldered on the opposite face. Electrical measurement with a VTVM was accomplished using this lead with one of the thermocouple leads. The sample was placed in a slotted refractory brick and the wires shielded with Fiberfrax. A direct torch flame on the sample was avoided by covering the sample with a stainless steel cover. Samples were subjected to 350°F cure for one hour prior to testing.

A "moderate" heating rate of 200-1600°F in 53 seconds was employed for the first test involving B147. A minimum resistance of about 2 megohms occurred at the lower temperatures. A value of > 5 megohms was observed at 1600°F. A second test with much more rapid heating, 16 seconds, revealed a minimum of about 0.55 megohms and ≯5 megohms at 1600°F.

Two runs were made with "H" cement, each consuming eight seconds. The minimums were 1.2 and 0.70 megohms; resistances higher than 5 megohms were obtained at 1600°F.

h. Vacuum Curing

Curing of the aluminum phosphate composition in vacuum (80-140 microns) indicated a reduction of curing time but no reduction of required

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Table V. ALUMINUM PHOSPHATE CEMENTS: AGING EFFECTS
ON RESISTANCE (megohms) VS. TEMPERATURE

Test:	A[1]		B[2]	
Formulation	"H" Cement	B147	"H" Cement	B147
200°F	750	200+	750	500
250°F	300	90	500	120
300°F	130	30	150	37
350°F	42	9.5	58	14
400°F	14.0	3.2	21	8.5
450°F	5.6	1.1	10.5	6.0
500°F	4.7	1.0	7.1	4.6
550°F	6.0	1.6	7.3	4.3
600°F	6.75	1.7	6.8	4.5
650°F	5.8	2.2	5.4	4.5
700°F	4.7	2.6	4.0	4.3
750°F	4.0	2.8	2.8	4.0
800°F	3.2	2.7	2.2	3.6
850°F	2.4	2.6	1.5	3.2
900°F	1.5	2.4	0.90	2.5
1000°F			0.40	1.7
1100°F			0.16	1.0
1200°F			0.060	0.65
1300°F			0.026	0.39
1400°F			0.013	0.21
1500°F			0.009	0.067
1600°F			0.006	0.039
Strength after test	825CA	75A	0A	150A

(1) A: Rapid heating rate of 200°-900°F in three to three and one-half minutes.

(2) B: Moderate heating rate of 200°-1600°F in about 30 minutes.

Note: All samples had the history: air dry for about 60 hours, 350°F/1 hr.

temperature. Vacuum curing appears to improve strength. As the main objective of this program, the reduction of curing temperature, was not served by this technique, vacuum methods were discontinued.

IV. SUMMARY AND CONCLUSIONS

Screening tests were developed for quick evaluation of commercial and experimental high temperature strain gage cements. Specimens were prepared by bonding two one-half inch diameter and half-inch high inconel cylinders. Rods were attached to these after curing for electrical leads in resistance measurements and for grips in tensile strength determinations.

Electrical measurements were conducted by inserting the specimen assembly into a tube furnace operated by a powerstat and temperature recorder-controller. More rapid heating was accomplished by suspending the specimen between quartz heating tubes.

Tensile strengths were obtained by mounting the specimen assembly in a Dillon Tensile Tester. Strength measurements were useful in evaluating thermal cycling properties, bond integrity, and the effect of additives in the formulations.

Water suspensions of rubidium di- and trisilicates were prepared and used as a binder for silica in cement formulations. Good electrical properties were obtained at low temperatures but these compositions increased rapidly in conductivity with temperature. Their actual curing temperature was 650 to 700°F.

Compositions were prepared using phosphoric acid as a binder with a view to obtaining low curing temperatures by means of a high vacuum. The use of vacuum reduced curing time but not temperature and did not prevent rapid absorption of atmospheric moisture when the specimens were returned to ambient conditions.

The additive study for monoaluminum dihydrogen phosphate cements encompassed a variety of inorganic oxides and salts. Incorporation of zinc compounds results in cements with good strength, adequate curing at 350°F, but rather poor electrical resistance at elevated temperatures.

Addition of magnesium oxide provides a formulation which has properties similar to those of ZnO compositions but which lacks shelf life. This shortcoming is obviated through utilization of more inert salts of

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magnesium, MgF_2 and $MgAl_2O_4$, but the improvement is accompanied by an inability to retain good high temperature resistance after heating to 1600°F. Although excellent electrical properties are achieved by employing either the nitrate or chloride of magnesium, the bonding power of the phosphate is nullified.

The most satisfactory additive developed by the investigation is yttrium nitrate hexahydrate. Although strengths of B144 are lower than those of the basic composition B85, high electrical resistances are displayed after a 350°F cure at the higher as well as moderate temperatures, and a further improvement in electrical behavior exists after heating to 1600°F. Adequate shelf life permits preparation of this cement as a ready-mix. Yttrium oxalate cements, while exhibiting properties comparable to those of nitrate formulations, do not possess indefinite jar life and their value is limited to preparation in kit form, i.e., solids and liquids separate.

Attempts to strengthen the yttrium nitrate compositions by addition of magnesia alumina spinel met with limited success; a slight gain in physical properties was accompanied by a mild decline in electrical resistances.

V. RECOMMENDATIONS

It is recommended that the final evaluations of the most promising experimental cements be performed using actual strain gage installations. The Aeronautical Structures Laboratory or the National Bureau of Standards could ably perform this testing. In the Foundation's test work, the resistance was measured through the cement film rather than its effect on an electrically conductive member such as a strain element. Therefore, the effect of an observed cement resistance on gage readings can only be assumed. As was noted in a previous section, a resistance of at least one megohm is believed to be necessary for a 100-gage installation. However, a momentarily lower resistance occurring after a low temperature cure may or may not upset gage measurements. Only application testing can ultimately determine how low in temperature the recommended cement may be effectively cured.

Therefore cement B144 having the composition:

100 g   Ottawa special microsil (silica)

  5 g   Chromic anhydride

  3 g   Gulton Alucer MA (alumina)

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    4 g   Yttrium nitrate hexahydrate

    35 cc   Victor Chemical monoaluminum dihydrogen

             phosphate (50 per cent)

    20 cc   Goulac suspension (1 per cent)

will be sent to the sponsor under separate cover for additional testing and use. Excellent electrical properties, effective curing at 350°F, and moderate strengths are displayed by this cement which is the most satisfactory formulation developed in the investigations.
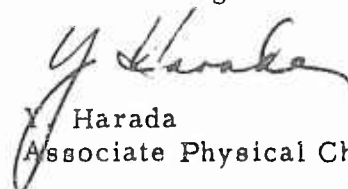
VI. LOGBOOK RECORDS

Detailed data are recorded in Logbooks C1162, C1165, C10434, C10437, C10440, C10748, C10750, C10752, C10755, C10758, C10759, C10762, C10767, C10769, C11339, and C11340.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION
of Illinois Institute of Technology

H. L. Rechter
Chemical Engineer

Y. Harada
Associate Physical Chemist

APPROVED:

S. W. Bradstreet
Supervisor
Inorganic Technology

Figure 1. SAMPLE PREPARATION

        (a) Uncemented Inconel Cylinders
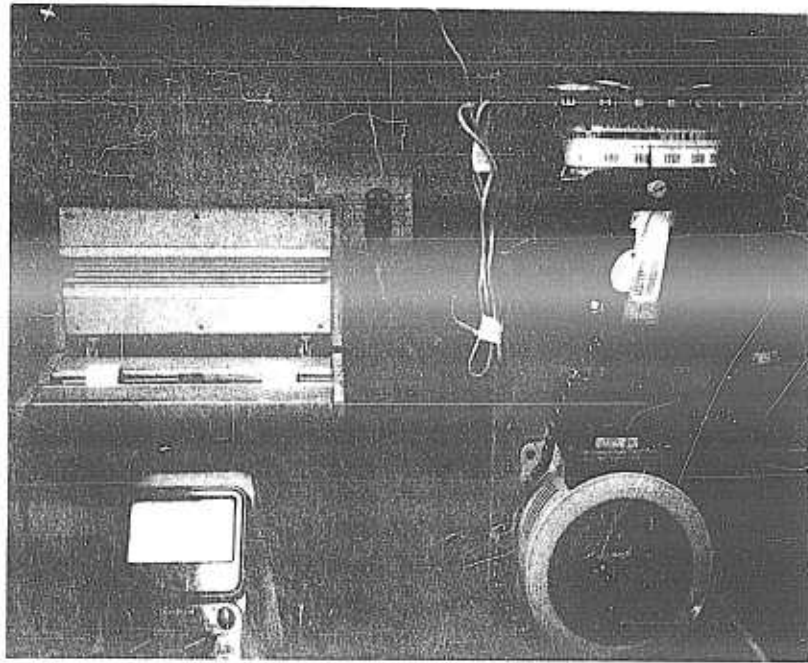
        (b) Cemented Sample

Figure 2.   EQUIPMENT FOR MODERATE
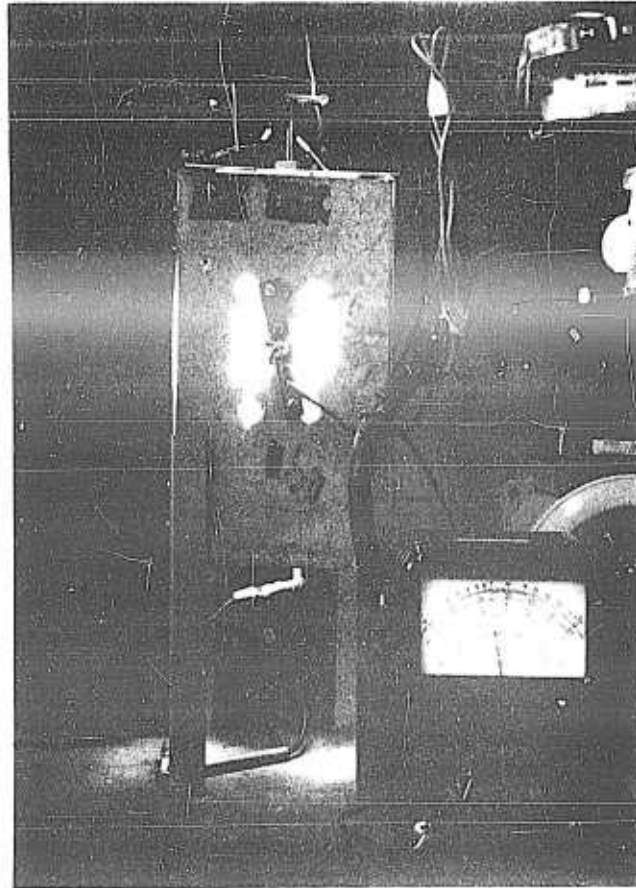   HEATING RATE TESTS
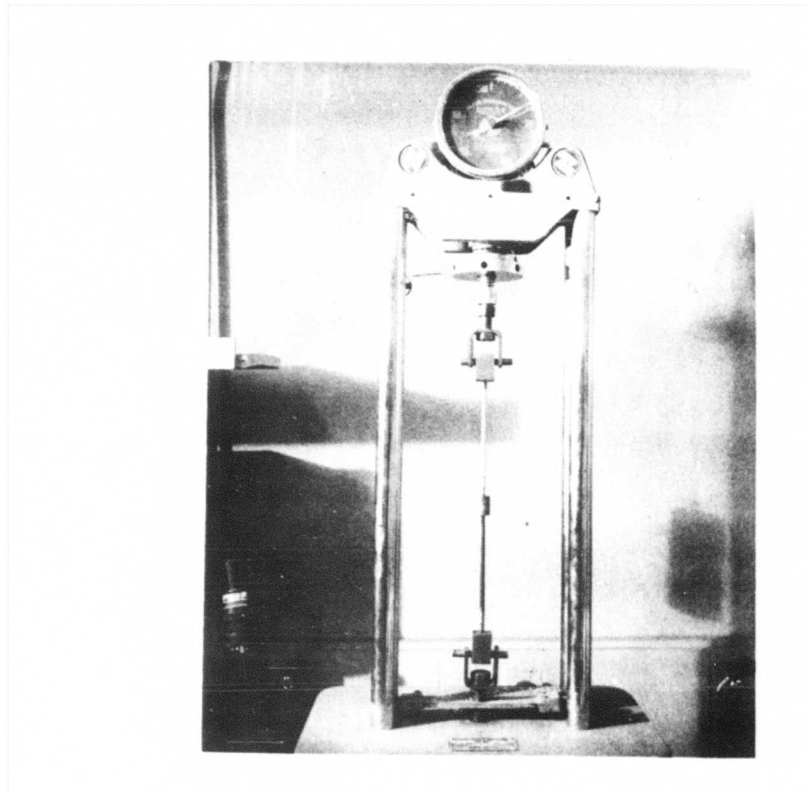
Figure 3. EQUIPMENT FOR RAPID
HEATING RATE TESTS
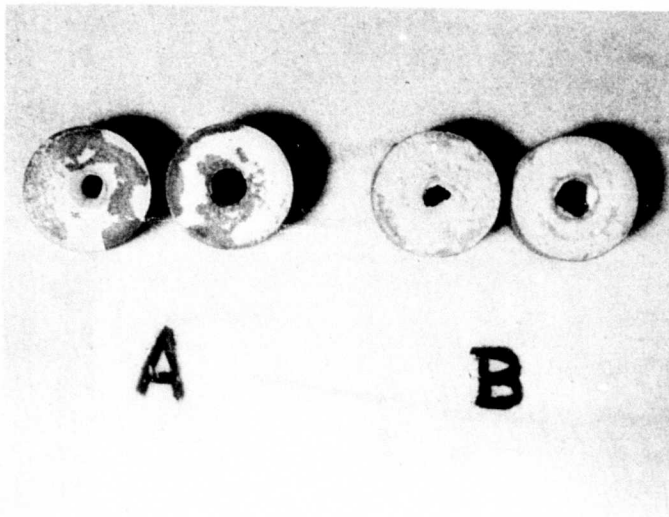
Figure 4.   APPARATUS FOR TENSILE STRENGTH TESTS

Figure 5. SAMPLE FAILURE IN STRENGTH TESTS
(a) Adhesive
(b) Cohesive

LEGEND FOR FIGURES 6 THROUGH 17

Curing temperatures are specified by the resistance-temperature points:

o   110°F

⊙   350°F

x   Temperature Indicated

Heating rates for the determination of resistance as a function of temperature are denoted by the curves:

———————   200-900°F in about nine minutes or

200-1600°F in about 28 minutes

- - - - - - - - - - - -   200-900°F in about three to four minutes

Each curve has a brief rotation of curing history plus the strength of the sample after the thermal cycle. Coding curves are depicted from 900°-800°F and 1600°F-1200°F.
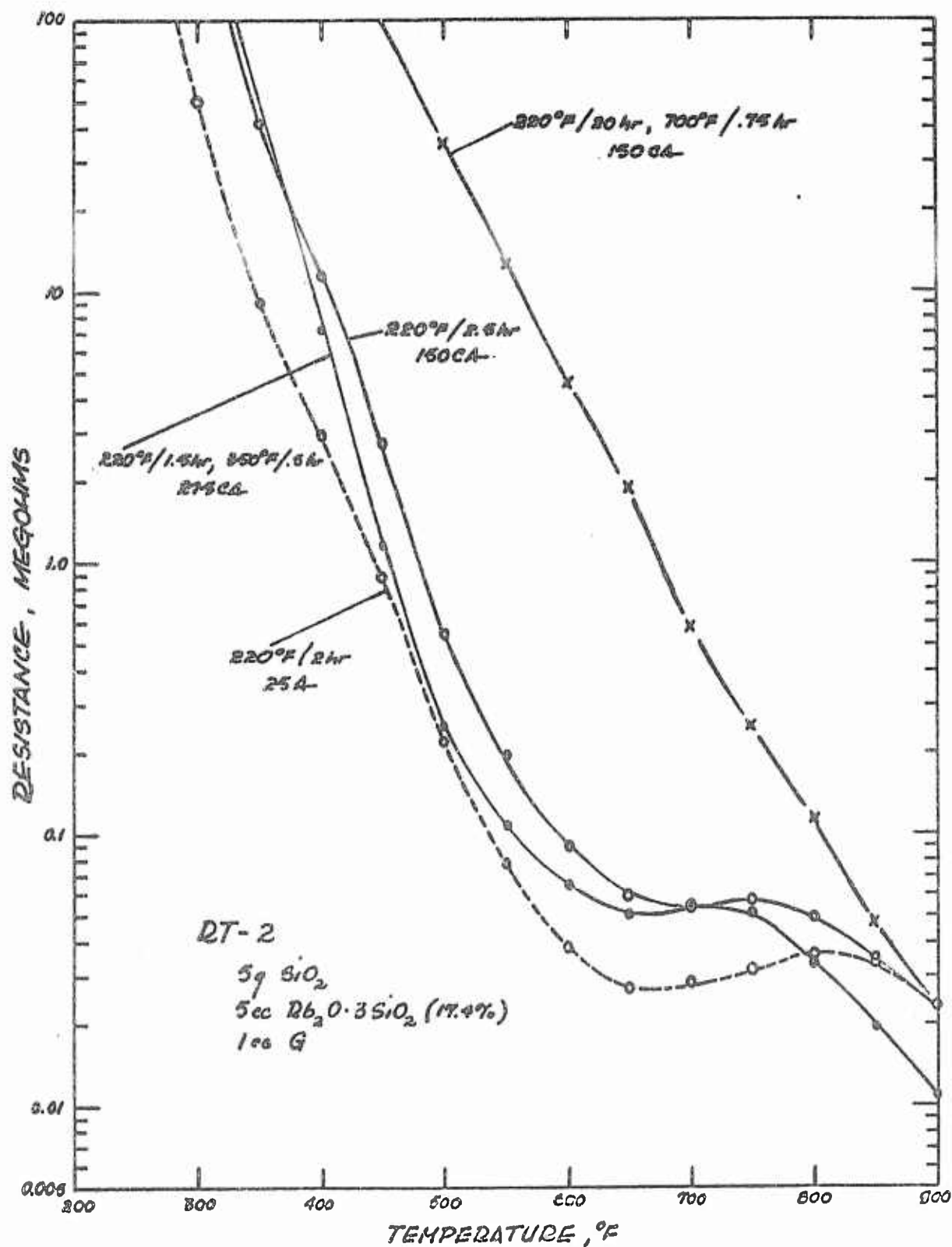
FIGURE 6. RT-2
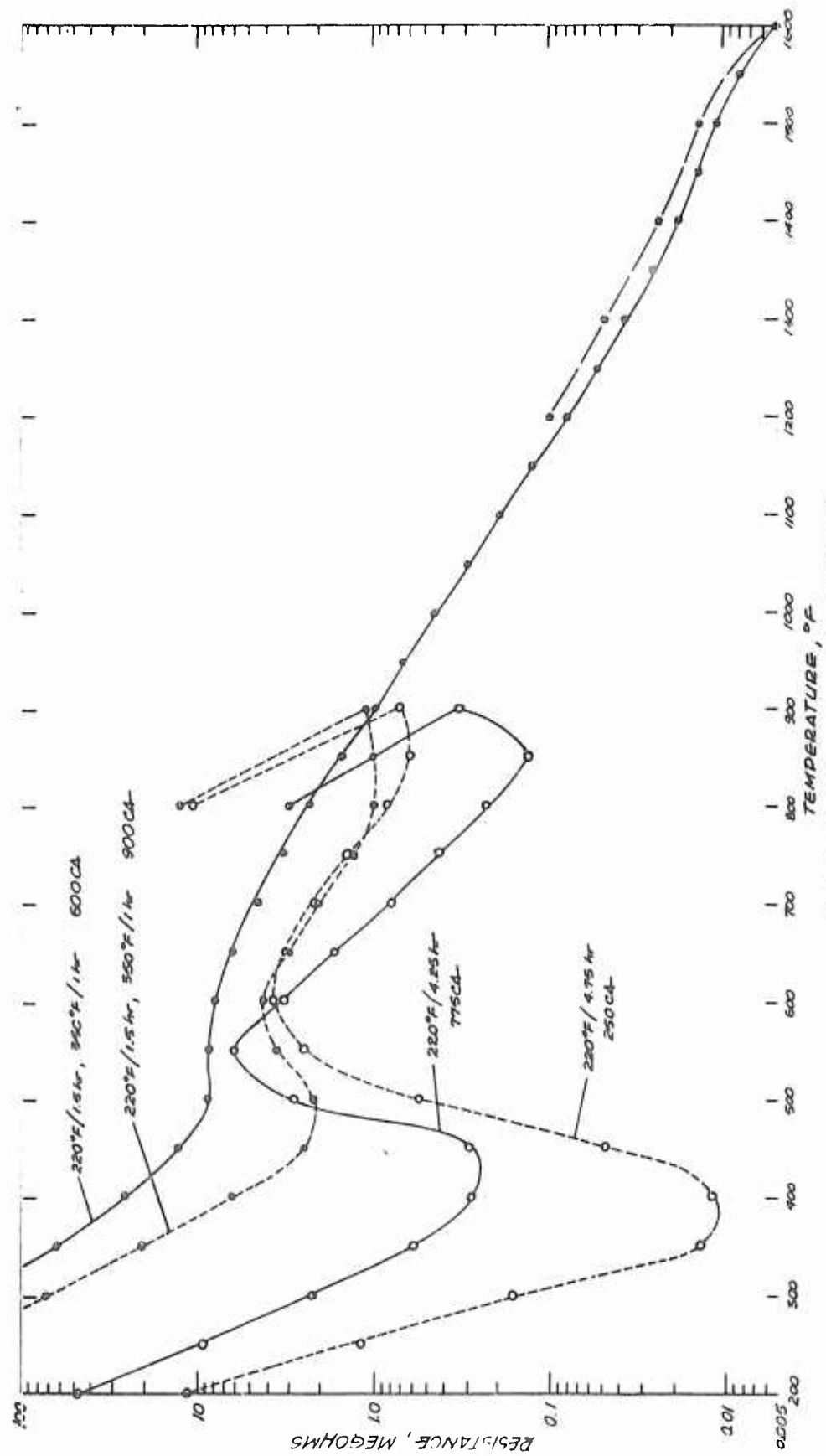
FIGURE 7. 4-CEMENT

FIGURE 8. B 85

FIGURE 9. B 82.

B 93
10g. SiO_2
0.5g. Cr_2O_3
0.5g. MA
3.5 cc AD
2.0 cc G
0.2g MgO

TEMPERATURE, °F
FIGURE 10 - B 93

DESISTANCE, MEGOHMS
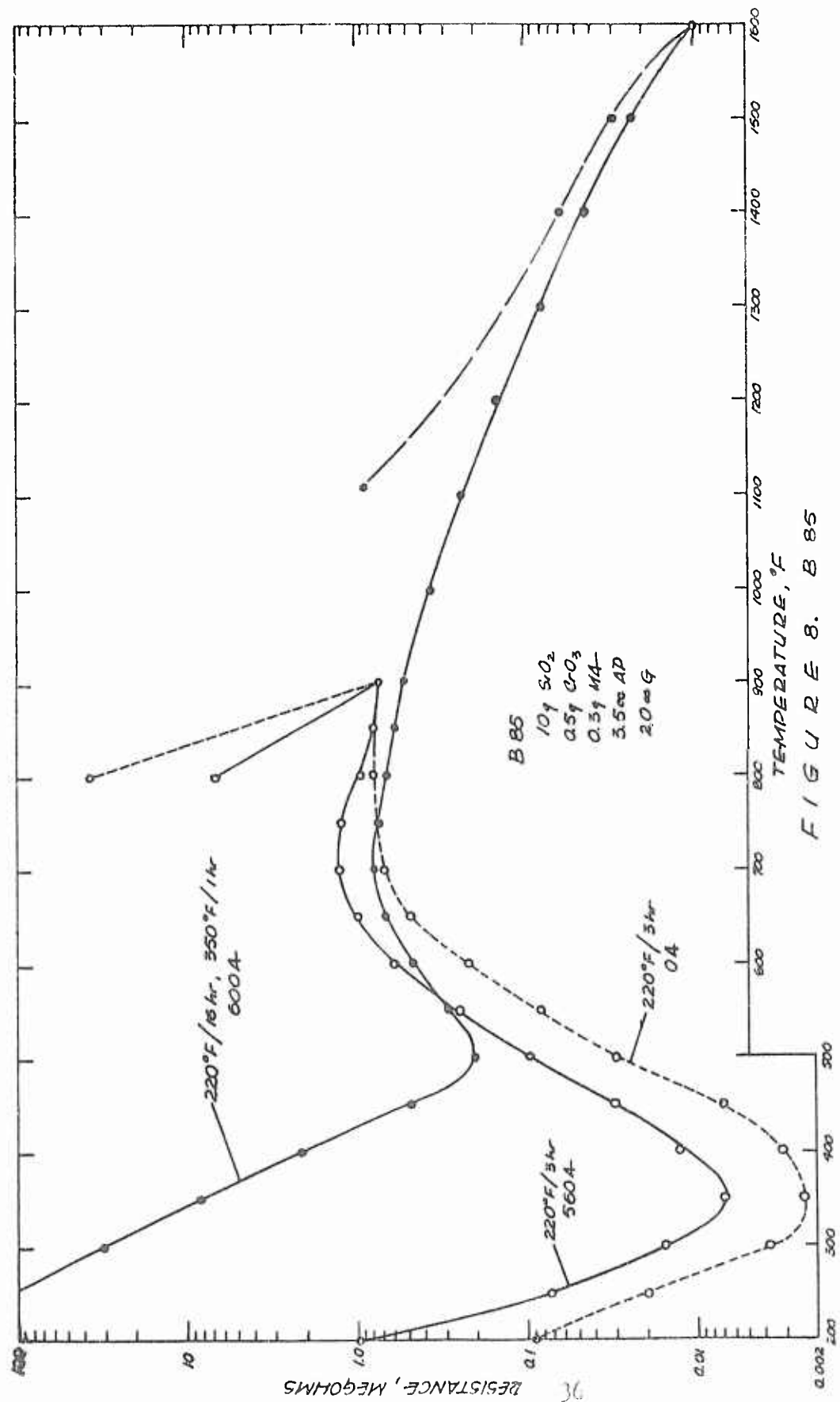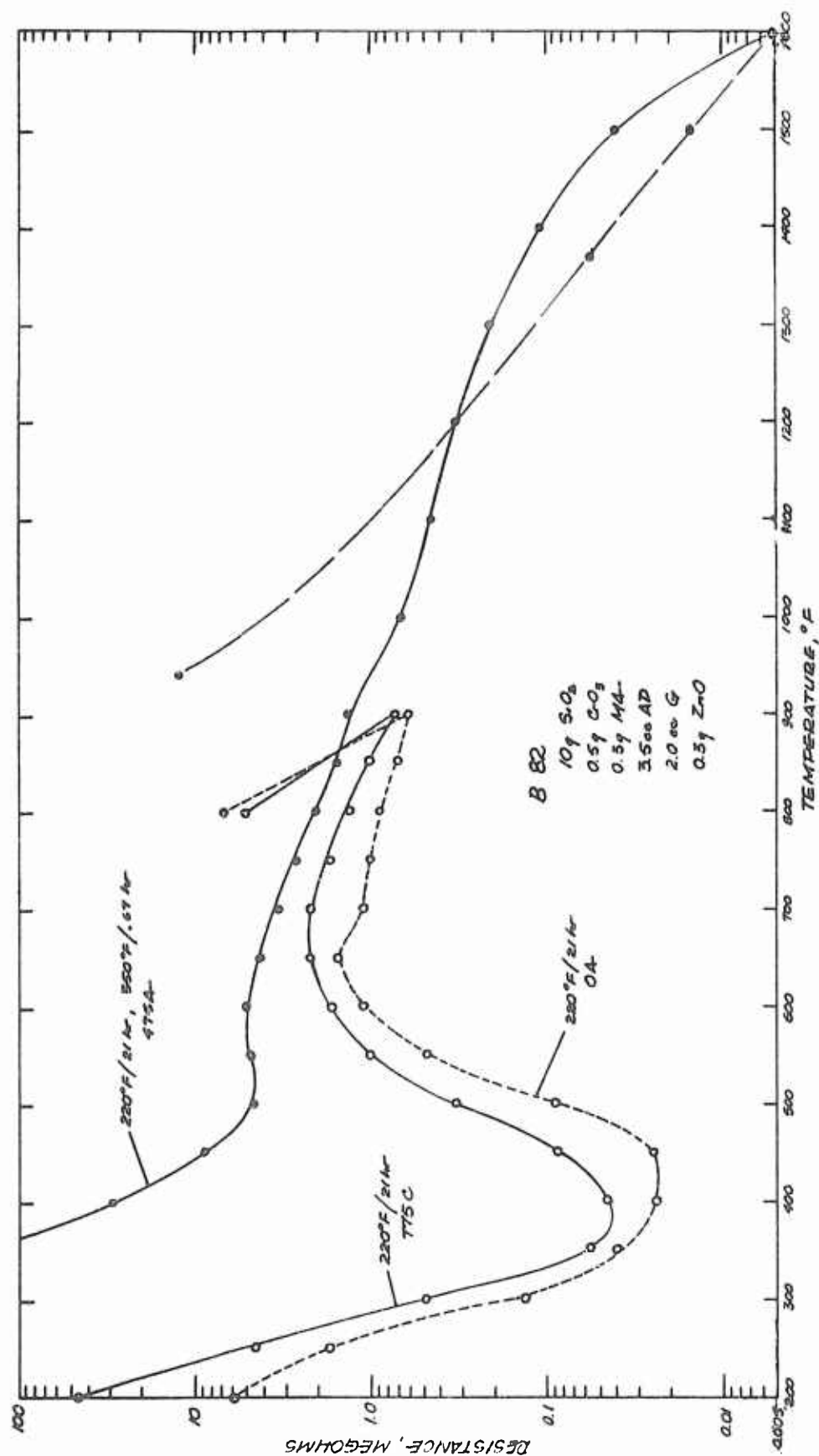
FIGURE 11. B127

FIGURE 12. B 134

B 134
10 g SiO₂
0.5 g CaCO₃
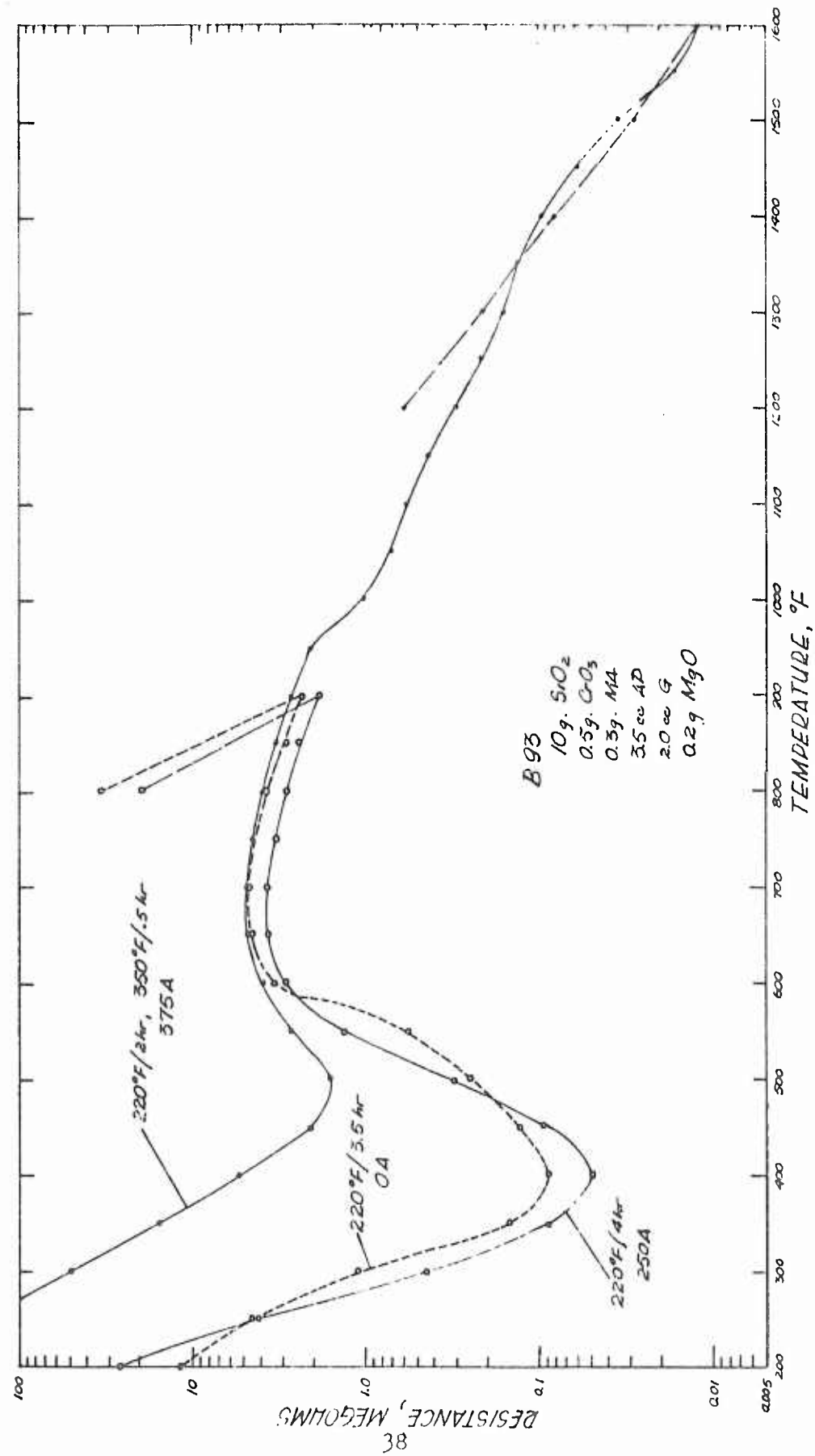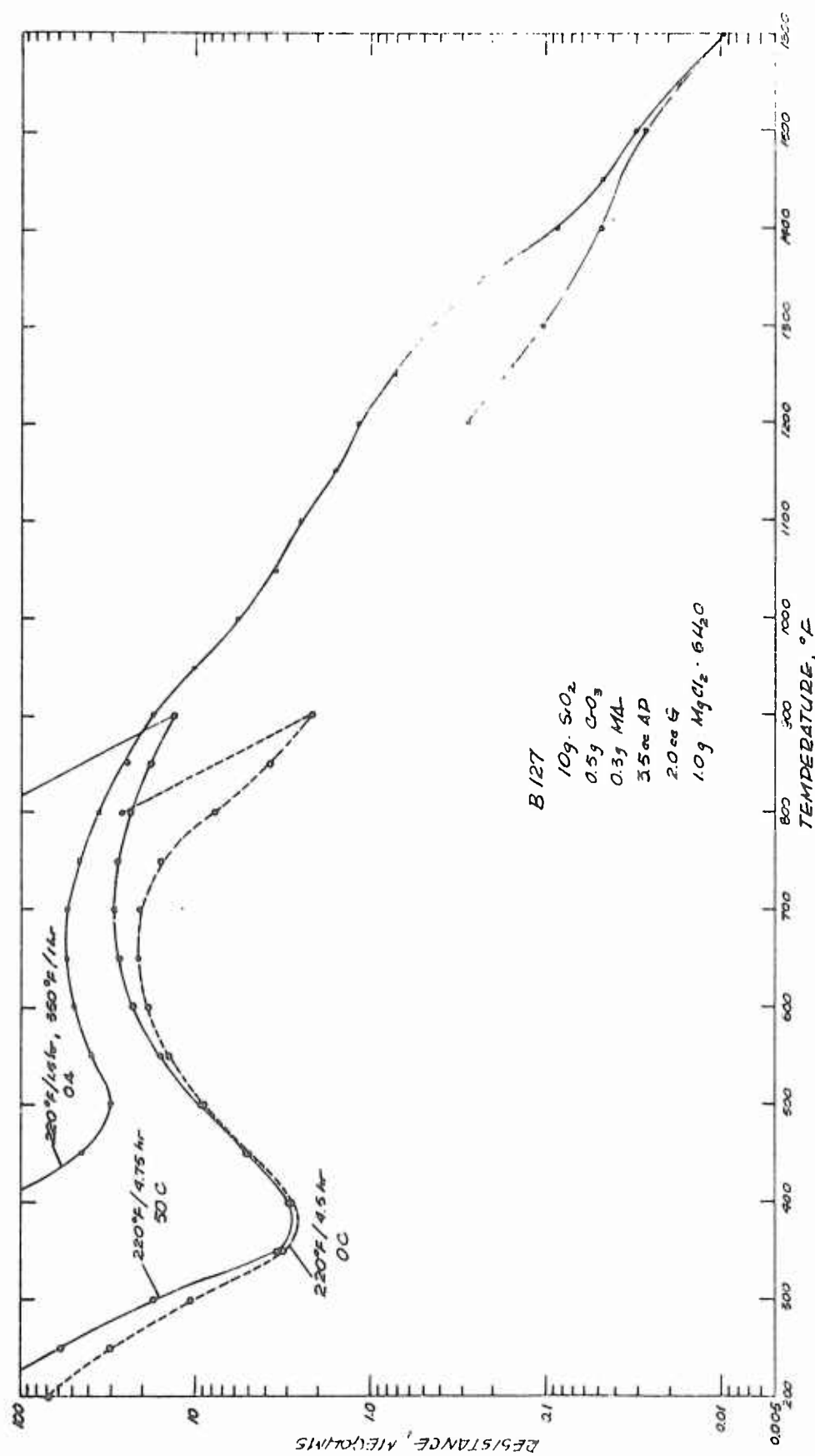0.6 MA
3.5 cc AP
3.0 cc G
0.4 g MgF₂
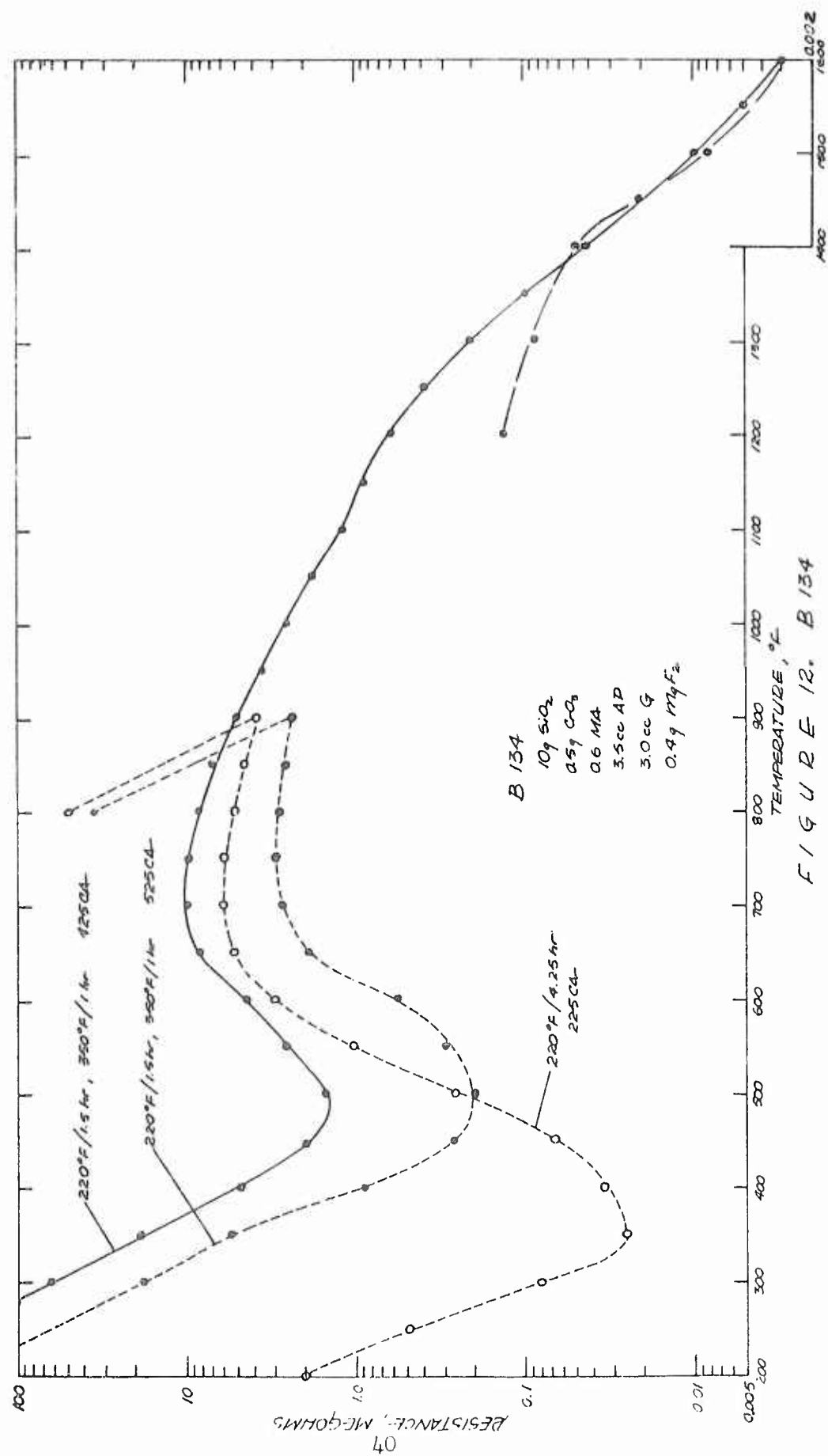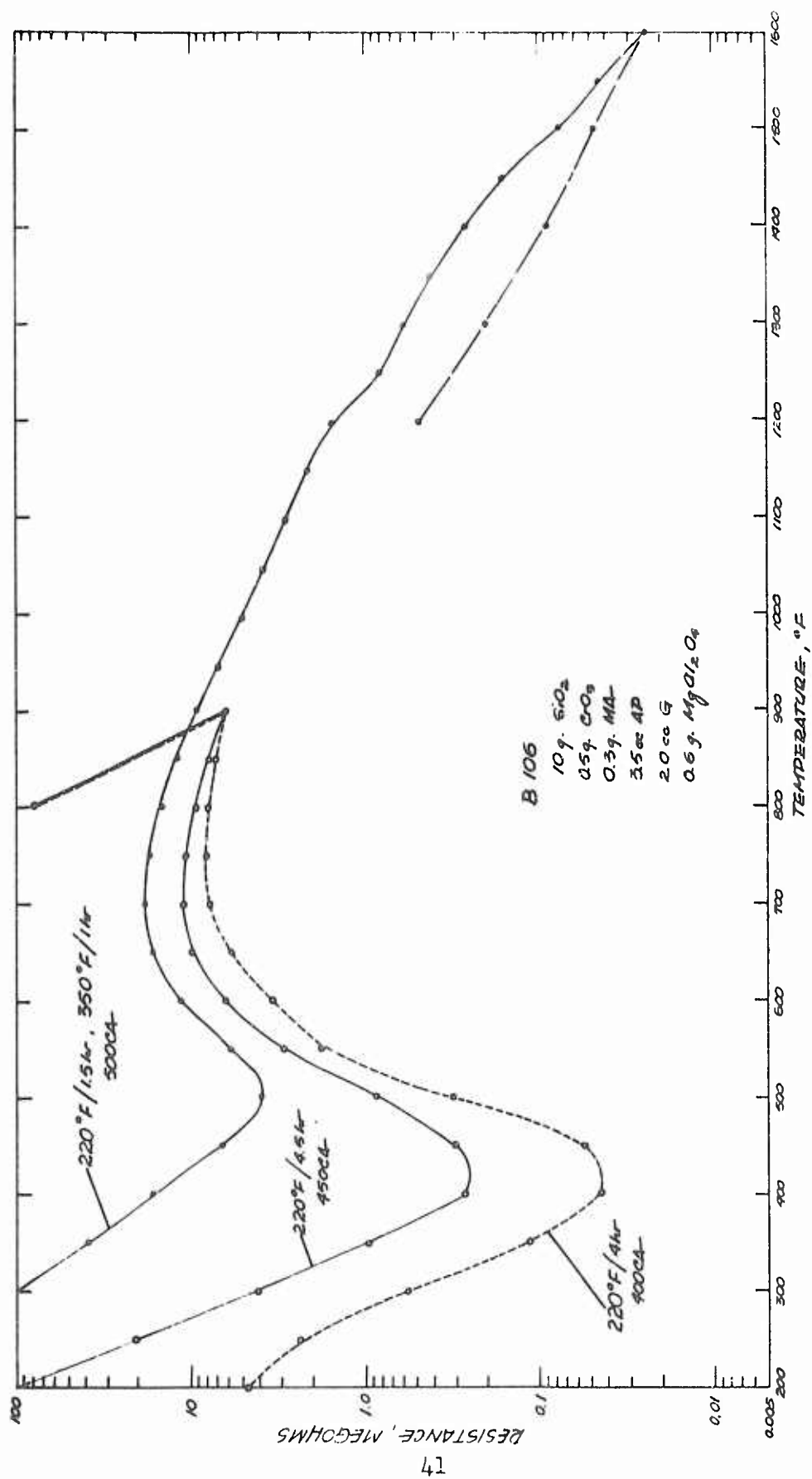
FIGURE 13. B 106
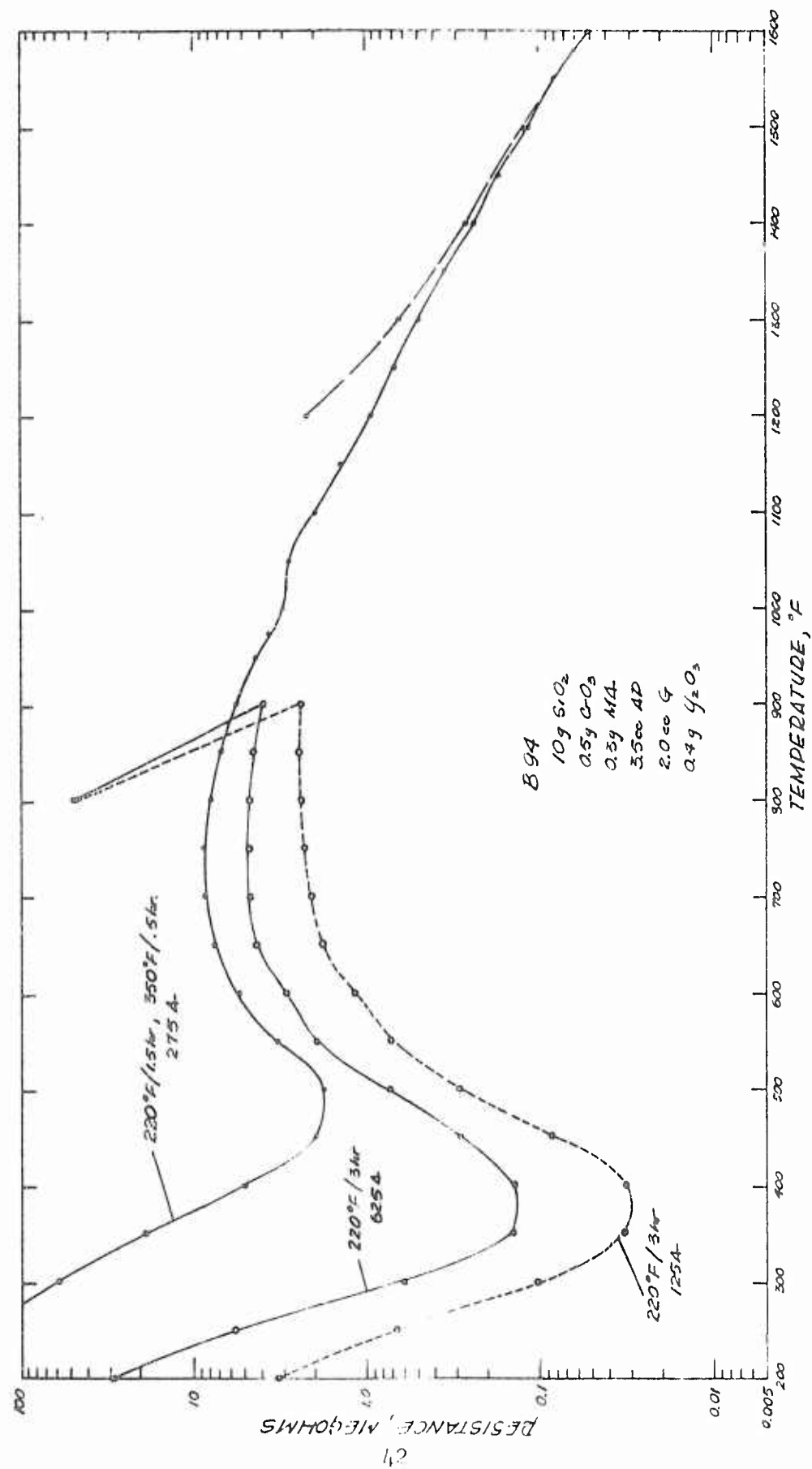
FIGURE 14. B 94
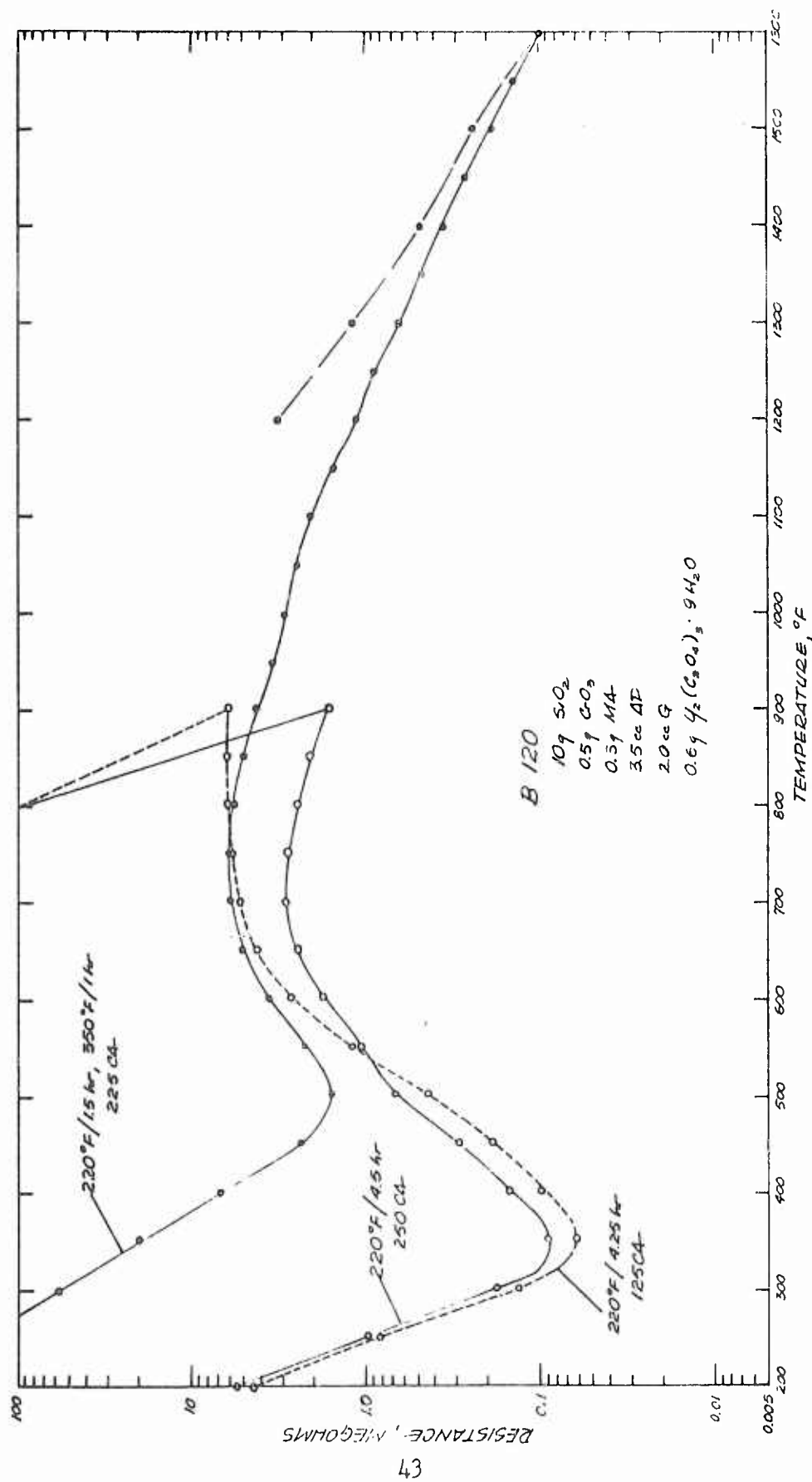
FIGURE 15. B 120
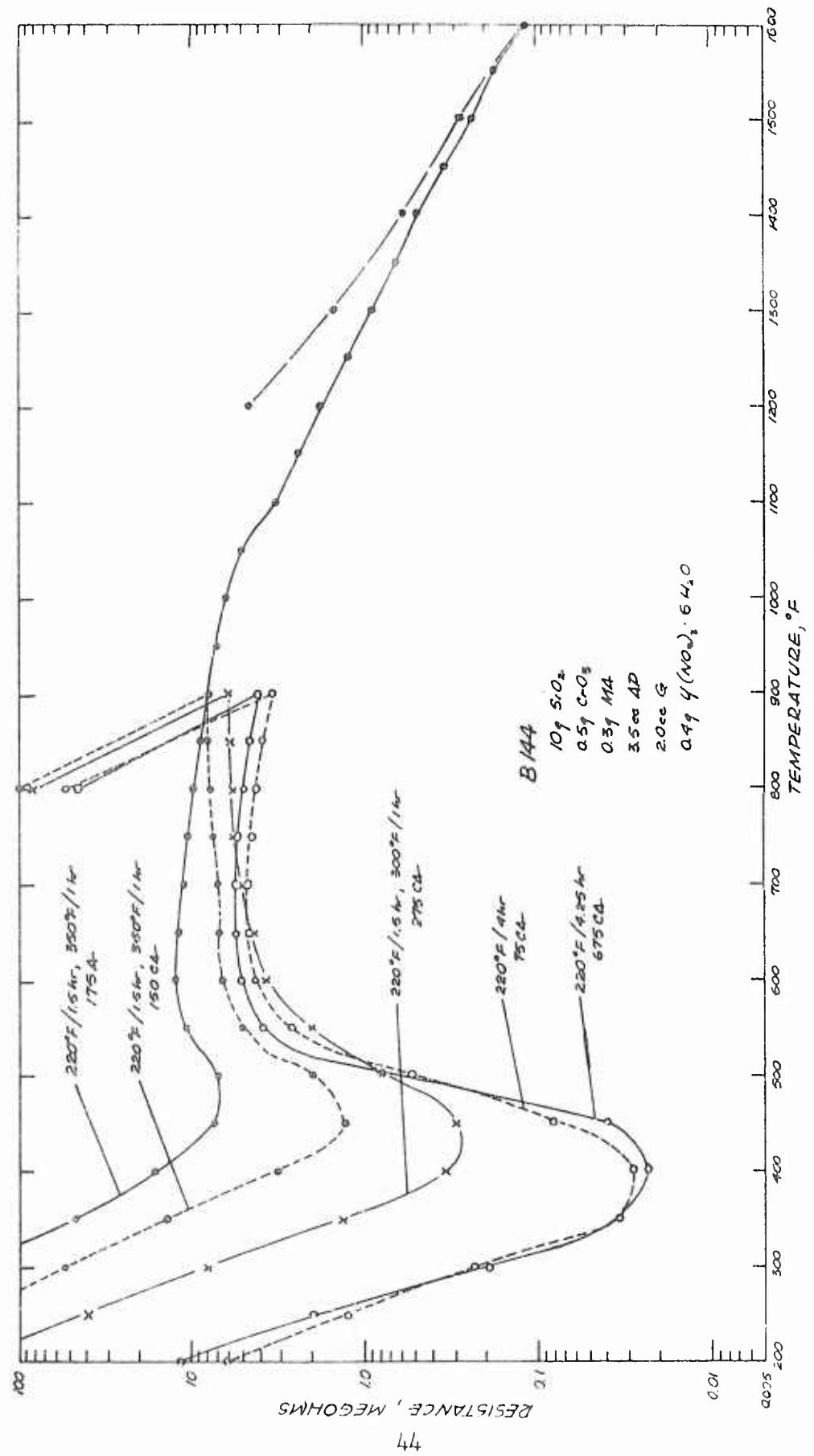
FIGURE 16. B 144
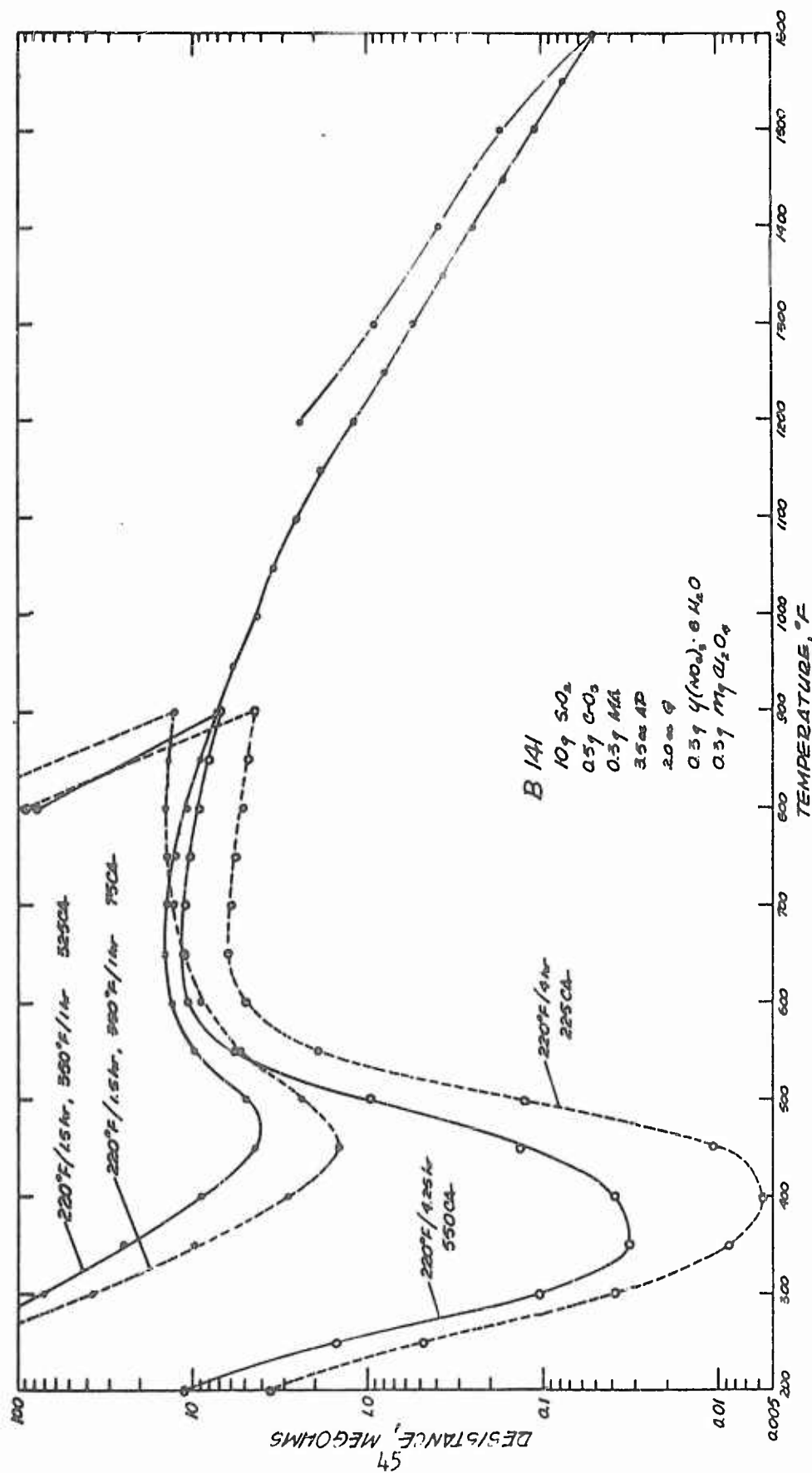
FIGURE 17. B 141

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Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

DEVELOPMENT OF HIGH-TEMPERATURE STRAIN-GAGE BONDING MATERIALS CURABLE AT LOW TEMPERATURES; by H.L. Rechter and Y. Harada, July 1961 (Contract No. N156-39304) - 49 p.

1. Report No. NAVAIRMATCEN-ASL-1050, Part V
2. P.A. No. 1-23-8(formerly project RAAD-298.1)

Investigations have been conducted to develop a high-temperature strain-gage cement which can be cured at low temperatures. Various compounds were incorporated into a basic formulation consisting of silica, alumina, chromic anhydride, monoaluminum dihydrogen phosphate, and Goulac suspending agent. The most satisfactory additive was yttrium nitrate hexahydrate which yields a composition, B144, with good curing at 350°F, excellent electrical behavior to 1600°F, and moderate strengths. Data on properties of cements containing other yttrium compounds, as well as zinc and magnesium compounds, are included in this report.

Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

DEVELOPMENT OF HIGH-TEMPERATURE STRAIN-GAGE BONDING MATERIALS CURABLE AT LOW TEMPERATURES; by H.L. Rechter and Y. Harada, July 1961 (Contract No. N156-39304) - 49 p.

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Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

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